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Oral Presentation Abstracts

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Understanding the Crystal Scale Performance of Structural Materials

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Mechanical design with engineering (structural) materials such as polycrystalline metallic alloys has been well-served by traditional methods for linking microstructure to mechanical properties using image-based data and mechanical tests of representative specimens. Regardless of the level of detail, however, microstructural data on their own contain no information regarding how the aggregate responds to loading; models for the mechanical response of an engineering alloy have no direct link to microstructure. Over the past decade, a suite of synchrotron-based High Energy x-ray Diffraction (HEXD) methods have been developed for measuring three-dimensional orientation maps that include every crystal within a polycrystalline aggregate. More importantly, the distortion of each crystal can be determined. Using *in situ* sample loading machines and high-speed area detectors, the evolution of every metallic crystal during elastic-plastic deformation of a test specimen can be observed. Due to crystal-scale anisotropies, each crystal experiences a unique stress and plastic deformation state even during simple uniaxial loading. Therefore the HEXD experiments can literally transform each diffraction volume into a single-crystal experimental testbed and the data from these experiments can be used to motivate and validate crystal-scale material models for alloy performance [1]. This talk describes some of the HEXD development that has taken place around the world over the past 10-15 years, focusing specifically on some of the instrumentation and software development that enables these experiments. Interfacing these data with the most advanced crystal-scale material models will also be described.

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Materials Discovery at High Pressures in Earth and Energy Sciences

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High pressure categorically alters materials and their physical, chemical properties, and impacts on all disciplines of materials science. Recent progresses in the brilliance, coherence, and spatial, energy, and temporal resolutions of high-energy synchrotron source have enabled a battery of powerful x-ray probes capable of penetrating the strong pressure vessels for *in-situ* characterization of samples under extreme pressures.

Our knowledge of the solid Earth is garnered through concerted research in petrology, geochemistry, geophysics, geodynamics, and mineral physics. Due to the extreme pressures and temperatures in the deep interior, all minerals go through drastic changes. Their structures, properties, dynamics, and chemical interactions must be simulated and investigated in laboratories under the realistic geotherm *P-T* conditions. Indeed, ample experimental advances have been made on the basis of the synchrotron x-ray diffraction, absorption, spectroscopy, and imaging techniques.

To recover the novel high-pressure materials metastably at ambient pressure far away from equilibrium is an exciting new approach in energy materials research. Mounting examples reveal that the combination of high pressures and low temperatures not only *brings* matter, but also *sustains* matter, very far away from equilibrium. In these studies, pressure provides a powerful means for continuously tuning the free energy of the system; x-ray photons excite systems into highly metastable states, and low temperature and chemical tuning prevent the system from reaching equilibrium. By varying these parameters, characterizing the dynamically compressed or stressed and electronically excited materials with time-resolved probes, transition mechanisms and energy landscapes can be revealed.

Industry Research Program at SPring-8

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SPring-8 has achieved steady industry utilization of 20% mainly via X-ray analytical method in these years. Of particular interest of industry is, however, whether an investment in research via Synchrotron Radiation(SR) will optimise value of the core application for innovative technology and products. Thus, the industry's demands for cutting edge utilization of light source characteristics such as pulse characteristics, coherence, low emittance, etc. shall be increasing more and more. To meet the demands practically, the novel scheme of Industry-Academia Alliance were created and implemented for the soft-matter industry. The key concept of the scheme is to create the accountable organization where a unit is composed of one academia and one industrial company to define the role in the advanced SR utilization.

In 2007, the 19 industrial companies formed Advanced Soft-materials Beamline Consortium with university professors and constructed the dedicated contract beamline, BL03XU; Advanced Soft-material in 2009[1]. The variety of needs & seeds brought from industry has galvanized research community and extended the range of SR application. Consequently, various industrial outcomes have been produced through the cooperation and competition among the member companies. One of the most successful case was a core contribution to a master brand "Energy Saving Tire" building by Sumitomo Rubber Industries, Ltd., Yokohama Rubber Co., Ltd. and Bridgestone Corporation.

The diverse activity of the Advanced Softmaterials Beamline Consortium has brought new vision of industry application and stimulated a movement to launch project-oriented beamline construction also based on the Industry-Academia alliance scheme. At this moment, the BL36XU Catalytic Reaction Dynamics for Fuel Cell and the BL28XU Advanced Basic Science for Battery Innovation are under construction. From the technical viewpoints, the development of the Pinpoint Structural Measurement[2] with Panasonic Co., Lmt. has contributed to the promotions of advanced industry applications. The details of the Industry-Academia Alliance scheme will be presented with the status and the perspectives on advanced industry application.

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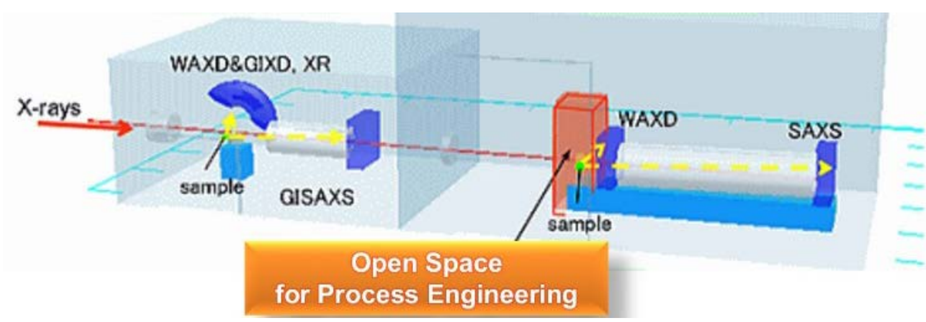

Intermediate-Energy X-ray Beamline for Soft X-Ray Scattering and Photoemission at the APS

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We have begun commissioning of a new, soft x-ray beamline for resonant soft x-ray scattering (RSXS) and angle-resolved photoemission (ARPES) at the Advanced Photon Source [1]. Its source is a 4.8 m long, electromagnetic undulator (EM-VPU) that provides horizontal, vertical or circular polarization in the range 250-2500 eV, and can run in quasiperiodic mode for harmonic suppression. A three-grating monochromator provides resolving a power of 2500, 10000, or 50000, depending upon the choice of variable line spacing (VLS) grating, with the corresponding incident flux ranging from 4×10^{12} to 10^{10} photons/sec. The RSXS and ARPES endstations reside on two different branch lines equipped with Kirckpatrick-Baez (KB) mirrors, providing a collimated (40 μrad × 80 μrad) beam for scattering and a focused (10 μm × 2 μm) beam for photoemission.

The RSXS endstation is equipped with a new capability that is unique in the world, namely a superconducting transition edge sensor (TES) array detector. This device has an intrinsic energy resolution of 1 eV and a quantum efficiency of 33%, which is ten times that of grating emission spectrometers. Comprising 240 pixels, this detector will provide a three orders of magnitude better signal than RIXS spectrometers at peer facilities.

In this talk, I will discuss our plans for bulk-sensitive ARPES studies of strongly correlated materials, and use of the TES detector for studies of charge order in superconductors, spin correlations in artificial spin ice, and quantitative structural analysis of polymer blends.

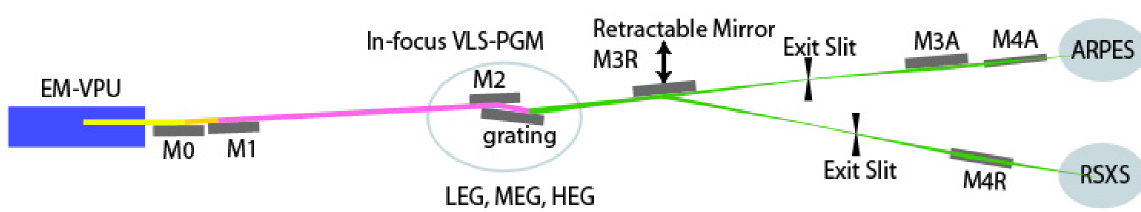

Figure 1 Schematic overview of the optical layout of the IEX beamline being built at Sector 29 of the Advanced Photon Source, Argonne National Laboratory. An EM-VPU with quasi periodicity capabilities produces variable polarization intermediate-energy x-rays (250-2500 eV). The focusing VLS-PGM has three gratings to deliver x-rays with high-flux and high energy-resolution into one of two dedicated endstations.

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Surface Diffraction with High-Energy X-rays at Beamline P07 at PETRA III

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The PETRA III High Energy Materials Science Beamline P07 is jointly operated by HZG and DESY. The DESY endstation is dedicated to fundamental research in science. One of the missions is the diffraction on surfaces and interfaces employing high energy X-rays between 50 and 200 keV taking advantage of the high penetration power and large Ewald sphere. For scattering on liquids a secondary optics to vary the incident angle on the sample surface is available.

The diffractometer is especially designed for surface diffraction [1] allowing precise sample alignment even in large, heavy complex sample surroundings for in situ studies like catalysis [2,3]. On the detector side a point detector and a flat panel 2D detector covering an area of 41 x 41 cm^2 are available. The detectors are placed on a long motorized translation, so that the distance to the sample can be varied between 0.5 m and 3.5 m. The beam can be focused by compound refractive lenses situated in an optics hutch in front of the experimental station. The distance between lenses and sample can be adjusted between 4.5 and 7.5 m. The size of the focus is up to 2 x 30 μm^2.

The concept of the secondary optics defining the scattering angle on liquid surfaces is following the design used at the ESRF [4], but here we employ two gradient crystals to increase the beam accpetance of the optics, which provides a stable beam on the sample.

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High Definition X-ray Fluorescence Imaging of Cultural Materials

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The X-ray fluorescence microscopy (XFM) beamline at the Australian Synchrotron utilizes three separate probes for its scanning operations: a zone plate nanoprobe, the workhorse KB mirror microprobe and a recently installed "milliprobe" for large area (1.1×0.6 m^2) elemental mapping.[1] The majority of the XFM studies have been performed with the large solid-angle 384-element energy dispersive Maia detector array.[2] The Maia detector routinely operates with pixel dwell times on the order of one millisecond, with full XRF spectra saved per pixel. The capability for imaging to around 10^8 pixels permits high definition large area mapping and 3D imaging modes, such as XANES imaging[3] and XRF tomography[4].

There has been an increasing desire of users to scan large "non-standard" specimens for scanning XRF imaging measurements ranging from 5-mm thick slices of human heart[5] to cultural materials such as paintings,[6] and historic objects such as the de Vlamingh pewter plate,[7] which have posed many new challenges for their analysis in a synchrotron setting. The XFM/Maia detector combination has proven effective for the analysis of cultural materials, because the large areas can be investigated in high definition while maintaining the context of the object, and the low dwell times minimize radiation dose to the sample. Recent studies involving the study of cultural materials and the approaches to their analysis will be discussed.

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In-situ Investigation of Metal/Polymer Interfaces by Soft X-ray Spectroscopies

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Interfaces between metal electrodes and semiconducting, π-conjugated polymers play an important role in polymer-based organic electronic and optoelectronic devices, such as organic light-emitting diodes (OLEDs), photovoltaic cells and field effect transistors. Generally, the interfacial chemical reaction and diffusion between metal electrodes and organic functional materials are considered to be detrimental to the organic-based optoelectronic device performance. In this presentation, we demonstrate that by controlling the interfacial chemical reaction to a certain extent, the device performance can be improved. Through in-situ investigation of the lithium thermal evaporation onto the poly (9,9-dioctylfluorene-co-benzothiadiazole) (F8BT) surface with synchrotron radiation soft X-ray spectroscopies, we found that although a strong chemical reaction occurs between Li and F8BT, by controlling the amount of lithium deposited, the interface dipole at the Li/F8BT interface can be tuned. Taking advantage of these Li/F8BT interfacial properties, we have fabricated F8BT-based prototype OLEDs by depositing different amounts of Li on F8BT first followed by depositing Al as the electrode. Indeed, through choosing an appropriate amount of Li to tune the interface dipole of Li/F8BT, an improved luminance and power conversion efficiency of F8BT-based OLED device can be achieved. These results for the first time demonstrate that the effect of interfacial chemical reaction and diffusion at the interface of metal electrode with functional organic material on the device performance cannot be generalized. These findings provide new insights into fundamental understanding of the relationship between the interfacial structures of metal electrode/organic functional material in organic (opto-) electronic device and the device performance, and will help to optimally design the organic electronic device structures with improved performances.

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Integrated Sample Environment for *Operando* Hard X-ray Spectroscopy

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Hard X-ray spectroscopy is a powerful tool to interrogate the structure and the electronic state of functional materials under in situ and operando conditions. Emerging methods, including high-resolution energy resolution fluorescence detection, emission spectroscopy and resonance scattering provide a wealth of information on the chemical system under working conditions, such as catalysts, batteries and fuel cells. The adoption of these tools by a wider community is constrained by several factors, including shortage of user-friendly facilities where necessary infrastructure, combining modern detection approaches and supplementary instruments to support operando experiments are available.

In this contribution we present the new approach where the sample photon delivery optics and detection systems are integrated with the sample environment and sample handling/ treatment system. This scheme is currently being developed for NSLS-II ISS (Inner Shell Spectroscopy) beamline at, slated to begin operations in the fall 2016. This tight integration allows sample illumination using polycapillary lenses, fluorescence collection at the large solid angles by means of SDD detectors, and detection of medium resolution X-ray emission signal with crystal spectrometers in two distinct geometries (von Hamos and spherical backscattering analyser). To allow for operando measurements, the gas handling system is designed to deliver a variety of gases and liquid vapours, relevant for catalysis and thin film deposition, to the sample during measurements; product analysis using mass-spectrometer is also implemented. The sample handling system will allow automated sample exchange and registration as well as off-line sample treatment, e.g., discharging of a battery cell while another cell is being characterized; these features are expected to significantly increase the throughput of the beamline.

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Combined Dry Plasma-etching and Online at-Wavelength Metrology for Manufacturing Highly-focusing X-ray Mirrors

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A new figuring station has been designed and installed at the ESRF BM05 which is the synchrotron beamline dedicated to instrumentation and methods development. This station aims at figuring mirrors with an elliptical shape capable of focusing X-ray beams down to spot sizes of a few tens of nanometers. Such an optical performance implies to generate mirrors with slope errors below 0.1 µrad rms, so that the surface height does not deviate from the stigmatic profile by more than a nanometer. Together with this requirement the process must preserve the surface roughness within the acceptable limit of 0.1-0.2 nm. In the production phase the time scale necessary for the figuring process should be of the order of a few days.

To meet such constraints, an iterative process has been developed to take advantage of at-wavelength measurements (i.e. using the X-rays themselves) for characterizing the mirror profile [1] before and after each shape correction step addressed using efficient ion beam sources [2]. A large vacuum chamber is hosting both the dry etching tool and the metrology instrumentation thus avoiding placement errors and surface contamination while reducing substantially the dead time between the profiling and metrology stages. In the ultimate process we start from a flat substrate and remove large amounts of material (few tens of micrometers). Nevertheless, the convergence to an elliptical shape takes only a few iterations.

This contribution will present the recent achievements obtained at the BM05 beamline regarding the on-line metrology method and the mirror profiles and lengths achieved.

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An Energy Dispersive Bent Laue Monochromator for K-edge Subtraction Imaging

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K-Edge Subtraction (KES) is a powerful synchrotron imaging method that allows the quantifiable determination of a contrast element (i.e. iodine) and matrix material (usually represented as water) in both projection imaging and computed tomography. With living systems, a bent Laue monochromator is typically employed to prepare imaging beams above and below the contrast element K-edge which focus at the subject location and subsequently diverge onto a detector. Conventional KES prepares the two beams by utilizing a splitter that blocks approximately 1/3 of the vertical beam size to prevent "edge crossing" energies beyond the monochromator.

A bent Laue monochromator has been developed that has very good focal and energy dispersive properties for KES. Approximately 4% of the vertical beam profile is involved in "edge crossing" energies, thus no splitter is employed. The beam can be narrowed vertically allowing a smaller crossover angle than a splitter based system which minimizes artifacts. The combination of good spatial resolution, energy dispersive properties, flux and a unique approach to data analysis make this system nearly ideal for KES.

Some of the relevant details of the monochromator will be discussed, especially the focal and energy dispersive properties, as well as, some details of artifacts caused by the beam focusing at the sample location. Example images of the beam and the object images will be presented as well.

Three-energy Focusing Laue Monochromator for the Diamond Light Source X-ray Pair Distribution Function Beamline I15-1

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The new wiggler side station beamline I15-1 at the Diamond Light Source will illuminate samples with a focused monochromatic X-ray beam to measure their atomic pair distribution functions. The monochromator will consist of three silicon crystals diffracting X-rays in the Laue case at a common Bragg angle of 2.83°. The first crystal will select 40 keV X-rays using the (1 1 1) planes, the second will select 65 keV X-rays using the (2 2 0) planes, and the third will select 76 keV X-rays using the (3 1 1) planes. The crystals will be bent tangentially to focus the selected X-rays onto the sample. All crystals will be cut to the same optimized asymmetry angle in order to eliminate image broadening from the crystal thickness. The bandpass, which will be dominated by the horizontal divergence of the wiggler source, will be tuneable up to 2%. Finite element calculations of the expected thermal distortion of the crystals will be provided and analysed to show the effects on the image size and bandpass. Preliminary X-ray topographs of the crystals have been taken to inspect residual strain from the fabrication process.

Design and Performance of a New Double-Laue Monochromator for High-energy X-rays at Cornell High Energy Synchrotron Source (CHESS)

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High-energy X-rays are efficiently focused in the out-of-scattering plane direction by sagittally-bent, asymmetric Laue crystals [1]. The main advantage of the Laue geometry over the Bragg geometry is the smaller beam footprint on the crystals and the ability to utilize the anticlastic bending in the meridional plane to eliminate the chromatic aberration of the beam by working on the Rowland circle [2]. A new double-Laue monochromator (DLM) optimized for high-energy X-rays has been installed and commissioned at CHESS. The DLM consists of several translation and rotation stages for alignment and tuning of energy, and a pair of sagittal benders, whose design is a significant improvement upon prototype benders that were previously fabricated and tested [3]. Several performance parameters such as flux, size of the focused beam and energy bandwidth ($\Delta E/E$) were characterized. The DLM, located 21 m from the source, focuses a 5 mm-or 20-mm wide (depending on the choice of aperture) 40 keV X-rays at the upstream crystal to approximately 1 mm FWHM at 27 m from the source. The X-ray energy ranges from 38 to 88 keV at a fixed vertical offset, with $\Delta E/E$ on the order of 10^{-3} and flux on the order of 10^{11} to 10^{12}, depending on energy. The DLM has fully remote-adjustable degrees of freedom for focusing and twisting on the both crystals. The heat load on the upstream crystal is handled with aluminum and graphite filters of various thicknesses and cryogenically-cooled gaseous helium flowing through copper tubes on the bender.

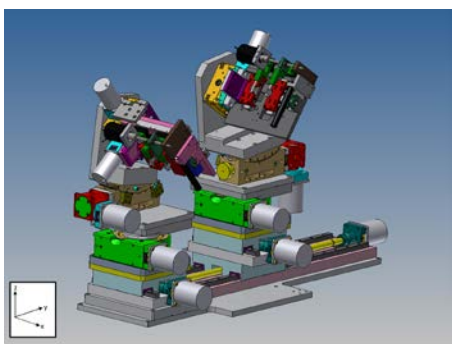

Figure 1: The DLM assembly, consisting of benders, and rotation and translation stages

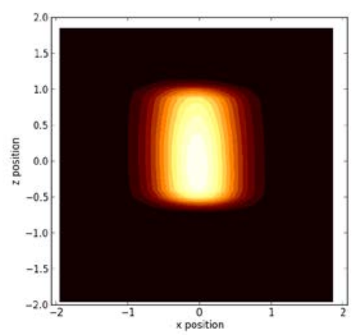

Figure 2: Intensity profile of the focused 40 keV beam

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Multilayer Gratings of the X-ray Monochromators of SOLEIL Beamlines for the 1 – 4 keV Energy Range

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Monochromatizing x-ray synchrotron radiation in the energy range centered on 2.5 keV is technically challenging. If a single layer coated reflective grating is used, the extremely grazing incidence and shallow groove depth which it requires make it difficult to ensure good performances. When looking for crystal diffraction, one is faced with the lack of good quality and stable crystals with large unit cell dimensions, and the large deviation angle requested by the available ones has a detrimental impact on stability.

It has be acknowledged that multilayer (ML) coated gratings can bridge the gap between the two technologies. The ML achievable periods of a several nanometers raise the deviation angles to a few degrees, while the penetration depth allows 20 or more layers to contribute to the reflectivity. To realize such gratings either the grating is etched first and the ML is deposited with a controlled period adapted to groove profile, either ML is deposited on a flat substrate and the grating pattern is etched in the ML. Both methods have been explored at SOLEIL and are respectively used at Deimos and soon Sirius beamlines, and at Lucia beamline for the etched ML type.

Each technology has its own advantages and drawbacks, leading to different application domains. It will be illustrated by the commissioning results achieved at these beamlines. They show that ML gratings are a viable way of extending upward the range of soft x-ray grating monochromators or, as well, extending downward to 1 keV the range of two-crystal monochromators.

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New Facility for Long Duration Experiments at Diamond Light Source

Claire A. Murray[a], *Annabelle Baker*[a], *Adam Bark*[a], *Tom Cobb*[a], *Sarah Day*[a], *Jon Kelly*[a], *Jonathan Potter*[a] *Stephen P. Thompson*[a], *Michael Wharmby*[a], *Guy Wilkins*[a], *Fajin Yuan*[a] & *Chiu C. Tang*[a]

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The high brightness beamline I11 at Diamond Light Source is a dedicated powder diffraction instrument which has been in user operation for a number of years. Equipped with multi-analysing crystals (MAC) and fast position-sensitive detectors, it is routinely used for high-resolution and time-resolved experiments [1-3]. Recently, a new facility for long duration experiments (LDE) has been added. Now in operation mode, this facility houses the necessary hardware and equipment for multiple LDE studies. These experiments are mounted on a large sample table equipped with adjustable linear drives to automatically and periodically move sample cells in and out of the beam and the Pixium area detector. LDE are set up and left in place with programmed automated data collections over periods of months or years. Sample environments such as electrochemical cyclers, incubators, heating stages, environmental chambers and high pressure gas cells are accommodated for user operation.

To complement the existing I11 facilities, the LDE hutch opens up new opportunities for those experiments which require weeks to months of periodically monitoring “slow” changes, up to two years. It is of particular benefit to research areas where important information on the development of phases over time cannot be obtained via ex-situ methods. Areas that will benefit include studies of crystallization, battery materials, gas storage, mineral evolution, seasonal effects, thermal and electrical cycling and corrosion science. With a versatile design and many automated features such as robotic sample changers, the upgraded beamline is used by many academic researchers from diverse scientific backgrounds and industries.

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Towards Ambient Pressure in the Micro- and Nano-materials Characterization by Scanning Photoemission Imaging and Spectromicroscopy

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Due to the short escape depth of electrons, less than a few nm, photoelectron spectroscopy is the best surface sensitive analytical techniques for probing surface and interface chemical composition and electronic properties. Nevertheless the standard approach to this technique suffers from two major limitations: spatial resolution, and the requirement for UHV conditions.

The Scanning PhotoEmission Microscope (SPEM) uses a direct approach to add the spatial resolution and characterize materials at the submicron scale i.e. the x-ray photon beam is downsized to a submicron spot and the sample surface is mapped by scanning the sample with respect to the focused beam. With the SPEM hosted at the Escamicroscopy beamline (Elettra-Sincrotrone Trieste) the beam can be downsized, by using Zone Plates, to a diameter of 120nm which allows imaging resolution of less than 50nm. The overall energy resolution is better than 200meV.

Only recent electron energy analyzer with differentially pumped lens systems allow to perform in situ XPS up to few mBar (near ambient pressure). Nevertheless due to their cost, technical complexity and low efficiency it was not possible to export such solution to photoemission spectromicroscopy so far. Results of innovative solutions developed for photoemission microscopes, based respectively on environmental cells with graphene or graphene oxide windows transparent to low-energy electrons[1], effusive cells where high- and low-pressure regions are separated by a small pinhole of 200µm diameter, and a dynamic controls of the amount of gas injected into the chamber toward the sample[2], will be presented and discussed.

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In-situ X-ray Nano-imaging Application in Energy Materials

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Transmission x-ray microscopy (TXM) is an emerging non-destructive full field hard x-ray nano-imaging technique. TXM provides x-ray nano-tomography capability to study microstructures in three dimensions (3D) which is critical to get full understanding of complex micro- and nano- structural information. Combining with the tenability of the x-ray wavelength, the TXM is also sensitive to elemental distribution and chemical states, which is ideally suited for in-situ chemical mapping of energy materials. Due to its non-destructive measurement, deep penetration, elemental and chemical sensitivity, and unprecedented nanometer scale resolution, TXM has been widely applied to the study of energy materials.

In situ observation of 3D microstructural evolution at the nanometer scale offers a direct way to look inside the electrochemical reaction of batteries to better understand the mechanism of structural degradation, to guide the engineering and processing of advanced electrode materials, and to produce accurate 3D parameters for theoretical simulations. The delithiation reaction in lithium ion batteries is often accompanied by an electrochemically driven phase transformation process. Tracking the phase transformation process at nanoscale resolution during battery operation provides invaluable information for tailoring the kinetic barrier to optimize the physical and electrochemical properties of battery materials. In this talk, we will present the structural and electrochemical changes at the nanoscale resolution in lithium-ion battery materials while the battery is cycling, using a newly developed TXM operating at the X8C beamline of the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL).

Real Time Investigation of Thin Film Growth in Sputter Deposition Processes

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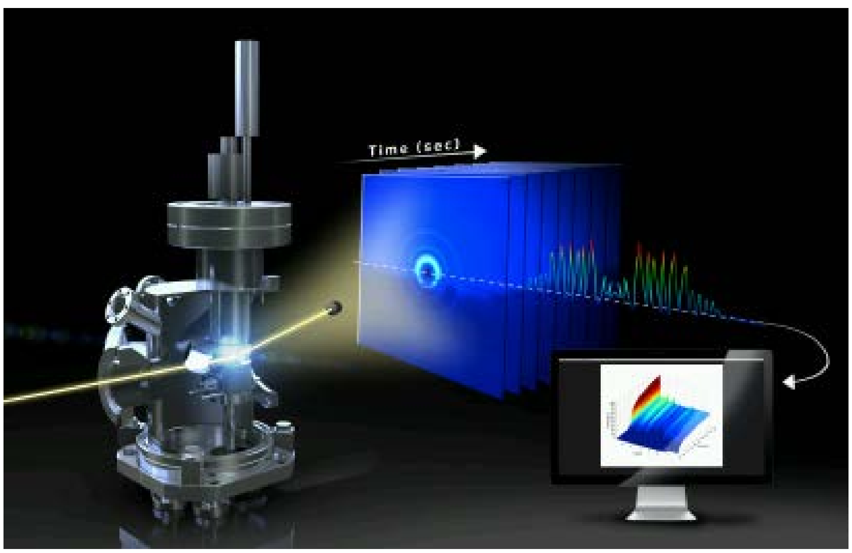
We have built a sputtering chamber that is suitable for the use at different synchrotron beam lines for in situ measurements [1]. In order to investigate thin films using the full capabilities of the synchrotron beam lines the sputtering camber is designed to be small enough to fit at different diffractometers. Therefore the size and weight of the unit is minimized without limiting the performance of the sputtering process. The goal is to investigate lead-free piezoelectric thin films. Structural and compositional control of the piezoelectric layers and the resulting electrical properties requires a fundamental understanding of the whole thin film formation process, from the first interactions at the substrate surface to the structure formation processes during film growth and crystallisation. The focus is on barium titanate ($BaTiO_3$), which is used e.g. in thin-film capacitors, non-volatile memories, electro-optical devices and MEMS devices [2, 3, 4]. The characteristics of the films strongly depend on the particular choice of processing parameters. Apart from this relation, this work is focused on the determination of the nucleation, the crystallisation pathway, and the microstructure evolution in $BaTiO_3$- layer samples. The presentation reports on results of measurements, the current status of the sputter chamber and in addition the next steps of development.



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Development of In-situ Sample Cells for Scanning Transmission X-ray Microscopy

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In-situ spectro-microscopic measurements with soft X-rays can reveal morphological, physical or chemical processes involving light elements. There are some technical challenges in soft X-ray microscopes, such as short working distance and requirement of vacuum. Scanning transmission X-ray microscopy (STXM) is a promising technique for in-situ observation, considering its characteristics, such as high energy and spatial resolution, low radiation damage and 2-dimensional chemical state analysis by using near edge X-ray absorption fine structure (NEXAFS). In UVSOR Synchrotron (Okazaki, Japan), the STXM system was installed in 2012 [1]. Since then several types of sample cells for in-situ measurement have been developed. One example is a liquid flow cell for in-operando electrochemistry. This consists of two 100 nm thick silicon nitride membranes sealed with O-rings. The liquid electrolyte flows in a few micron gap between these membranes, driven by a peristaltic pump from outside of the STXM chamber via a feed through. Three electrodes (50 nm thick gold) are deposited on one of the silicon nitride membranes. In addition to the in-situ flow electrochemical cell, we will show some other in-situ cells, including an azimuthal rotatable sample cell for polarization dependent measurements and a sample cell with humidity control. Results of applications of these in-situ devices will be presented

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Initial Performances of First Undulator-Based Hard X-Ray Beamlines of NSLS-II Compared to Simulations

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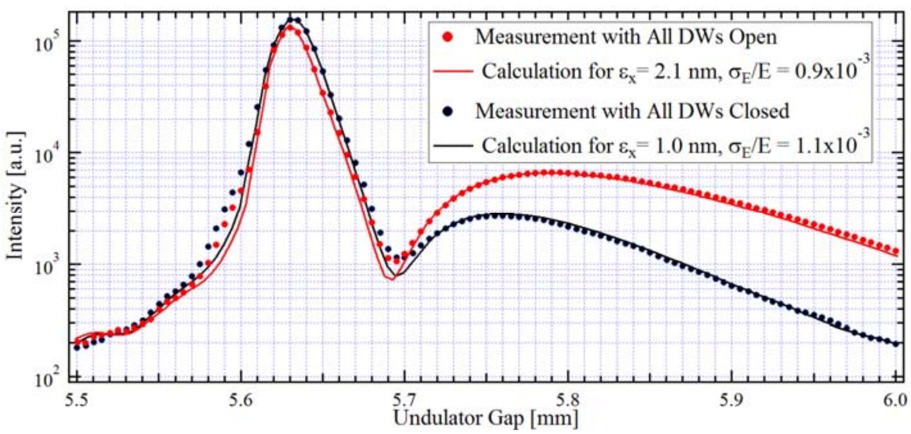
Commissioning of the first X-ray beamlines of NSLS-II included extensive and detailed measurements of spectral and spatial distributions of radiation beams at different locations of the beamlines, from front-ends to sample locations. Comparison of some of these measurements results with high-accuracy calculations of partially-coherent synchrotron (undulator) emission and wavefront propagation through elements of X-ray transport optics, performed using "Synchrotron Radiation Workshop" (SRW) code, will be presented. In some cases such comparison allows for diagnostics of electron beam emittance-related parameters as well as characterization of imperfections of beamline optical elements; in others, only a final deviation of a beamline performance from an ideal case can be evaluated. However, in all cases such comparison greatly helps to characterize beamline performances and determine potentials and directions for further improvement. In particular, the measurements and simulations show that hard X-ray undulator based beamlines of NSLS-II in general benefit from the reduction of the electron beam horizontal emittance from ~2 nm to ~1 nm thanks to the use of damping wigglers in the storage ring, though in some cases the quality of mirrors used for transporting X-ray beams to samples limits the associated final performance gains.



Example: undulator (20 mm period, 3 m long) radiation "gap spectra" at ~8 keV photon energy (5th harmonic) measured through a "pinhole" using an ion chamber detector at the Hard X-ray Nanoprobe beamline, with open and closed gaps of damping wigglers, and the corresponding SRW calculations for the "best-fit" electron beam parameters.

Performance of the P04 Online Diagnostic Unit for SR and FEL Radiation

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Modern synchrotron radiation beamlines such as the Variable Polarization XUV Beamline P04 at PETRA III [1] offer a broad set of user selectable parameters such as photon flux, energy, bandpass and polarization in order to provide optimum conditions for a broad range of scientific applications. However, for a successful experiment the given conditions should be precisely controlled and thus ideally be permanently monitored. Even more challenging is the situation at free-electron lasers where many experimentally relevant photon properties usually fluctuate on a shot-by-shot basis. In order to suit this needs we have developed an online diagnostic unit which can monitor relevant photon properties using a measurement principle which is also well-suited for FEL radiation.

During the past years, this diagnostic unit developed for the Variable Polarization XUV Beamline P04 at PETRA III has proven to be a valuable tool for beamline commissioning and characterization. The device employs angle resolved electron spectroscopy making use of 16 independent time-of-flight MCP-based detectors. It utilizes a dilute gas-phase target allowing an online determination of various photon parameters, such as degree of polarization, energy, bandpass and beam position, while the user experiment can record data with basically unchanged photon flux.

We will describe the basic principles of the device on the basis of the results obtained at different SR sources and will discuss the boundary conditions of the method. Selected examples from FERMI [2], FLASH and LCLS will be given for shot-by-shot online analysis highlighting its use also for beamline characterization and tuning.

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Transmission Diamond Imaging Detector

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Many modern synchrotron techniques are trending toward use of high flux beams and/or beams which require enhanced stability and precise understanding of beam position and intensity from the front end of the beamline all the way to the sample. For high flux beams, major challenges include heat load management in optics (including the vacuum windows) and a mechanism of real-time volumetric measurement of beam properties such as flux, position, and morphology. For beam stability in these environments, feedback from such measurements directly to control systems for optical elements or to sample positioning stages would be invaluable. To address these challenges, we are developing diamond-based instrumented vacuum windows with integrated volumetric x-ray intensity, beam profile and beam-position monitoring capabilities. A 50 µm thick single crystal diamond has been lithographically patterned to produce 60 µm pixels, creating a >1kilopixel free-standing transmission imaging detector. This device, coupled with a custom, FPGA-based readout, has been used to image both white and monochromatic x-ray beams and capture the last x-ray photons at the National Synchrotron Light Source (NSLS). This technology will form the basis for the instrumented end-station window of the x-ray footprinting beamline (XFP) at NSLS-II.

Fast Multi-Wavelength Photon Detector Based on Quantum Well Devices and Charge-Integrating Electronics for Non-Invasive FEL Monitoring

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The recent evolution of free-electron lasers has not been matched by the development of adequate beam-monitoring instrumentation. However, for both experimental and diagnostics purposes it is crucial to keep such photon beams under control, avoiding at the same time the absorption of the beam and the possible destruction of the detector. These requirements can be fulfilled by utilizing fast and non-invasive photon detectors operated *in situ*, upstream from the experimental station.

From this perspective, sensors based on Quantum Well (QW) devices can be the key to detecting ultra-short light pulses. In fact, owing to their high electron mobility, InGaAs/InAlAs QW devices operated at room temperature exhibit sub-nanosecond response times. Their direct, low-energy band gap renders them capable of detecting photons ranging from visible to X-ray. Furthermore, the 2D electron gas forming inside the QW is responsible for a charge amplification mechanism, which increases the charge collection efficiency of these devices.

In order to acquire the signals produced by these QW sensors, a novel readout electronics has been developed. It is based on a high-speed charge integrator, which allows short, low-intensity current pulses to be read within a 50-ns window. The integrated signal is acquired through an ADC and the entire process can be performed at a 10-MHz repetition rate.

This work provides a detailed description of the development of the QW detectors and the acquisition electronics, as well as reporting the main experimental results, which show how these tools are well suited for the realization of fast, multi-wavelength beam monitors.

A Phase Space Beam Position Monitor for Synchrotron Radiation

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Synchrotron radiation experiments critically depend on the stability of the photon beam position. The position of the photon beam at the experiment location is set by the electron beam source position and angle as it traverses the magnetic field of the bend magnet or insertion device. An ideal photon beam monitor would be able to measure the photon beam's position and angle, and thus infer the electron beam's position in phase space. Monochromatic x-ray beams at synchrotrons are typically prepared by x-ray diffraction from crystals usually in the form of a double crystal monochromator. Diffraction couples the photon wavelength or energy to the incident angle on the lattice planes within the crystal. The beam from such a monochromator will contain a spread of energies due to the vertical divergence of the photon beam from the source. This range of energies can easily cover the absorption edge of a filter element such as iodine at 33.17 keV. A vertical profile measurement with and without the filter can be used to determine the vertical angle and position of the photon beam.

The goal was to investigate the use of this system as a phase space beam position monitor. The system was tested for sensitivity to position and angle under a number of synchrotron operating conditions. The results are comparable to other methods of beam position measurements and indicate that such a system is feasible in situations where part of the white synchrotron beam can be used for the phase space measurement.

Investigations of Materials under Extreme Conditions of Pressure, Temperature, Ionization and Electro-magnetic Field at European XFEL

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The High Energy Density Science (HED) instrument at the European X-ray Free-Electron Laser Facility in Hamburg, Germany, is dedicated to the investigation of a wide range of materials and systems at extreme conditions of pressure, temperature, ionization or electro-magnetic field leading often to non-reversible states of the probed material. For sample excitation a variety of high energy drivers will be installed at this instrument [1]. Three separate optical laser systems will be available for very high time-resolution, dynamic compression and relativistic laser-matter interaction type of experiments. These drivers will allow studying various phase space parameters with time-resolution down to 10 fs, pressures into the TPa regime, and electric field strength up to 10^{20} W/cm^2. In addition, a pulsed-magnet will provide magnetic fields up to 50-60 T.

This unique instrument is designed to enable the application of various x-ray probes including spectroscopic, diffraction and imaging methods [2]. It will operate in the photon energy range from 3 to above 20 keV and will feature a variety of platforms facilitating the usage of different techniques in user-driven experiments. Due to the often non-reversible extreme excitation of the samples automated sample replacement schemes will have to be employed. Applications at this instrument will include condensed-matter physics, materials science, high pressure, planetary and plasma physics related problems.

Being on the baseline instrument of European XFEL first user experiments are planned for 2017. The installation of the high energy and high intensity lasers as well as of the pulsed magnets will be available through contributions by the Helmholtz International Beamline for Extreme Fields (HIBEF) User Consortium [3].

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Novel Portable Press for Synchrotron Time-resolved 3-D Micro-imagining under Extreme Conditions

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Synchrotron X-ray microtomography is a non-destructive 3D imaging/microanalysis method selective to a wide range of properties such as morphology, density, chemical composition, chemical states, structure, and crystallographic perfection with extremely high sensitivity and spatial resolution.
To extend this technique to extreme conditions (high-pressure/high-temperature/high stress), we developed at ESRF on beamline ID27 a new portable device, the RoToPEc [1], based on the Paris-Edinburgh press, where two opposed conical anvils are used to pressurize a sample embedded in an X-ray transparent boron epoxy gasket. In our new system, both anvils can rotate independently under load, with no limitation in angle, through two sets of gear reducers and thrust bearings. The accurate and simultaneous rotation of the top and bottom anvils is achieved using stepper motors and optical encoders positioned precisely on the both anvils. The ability to fully rotate the sample chamber under extreme conditions (up to 15 GPa and 2200K), overcomes the usual limited angular aperture of ordinary high pressure set-ups, allowing complete sets of tomographic projections to be acquired, in both full-field imaging (where a large (approx. 2x2 mm^2) monochromatic (or pink) x-ray beam is used to collect 2D radiographs) or micro-diffraction modes (scanning with a pencil beam of FWHM 3 x 3 μm^2 at several projection angles).

Additionally, independent and controlled rotation of each anvil enables operation in shearing (one anvil rotates while the other is stationary) or deformation modes (both anvils rotate in opposite directions) under high P,T conditions. Hence, our portable device can operate in four different modes: (i) tomography, (ii) shearing, (iii) deformation or (iv) combination of (iii) or (ii) and (i). Our portable device has been easily and successfully adapted to various multi-modal synchrotron experimental set-up at beamlines ID27 (**ESRF**), PSICHE (**SOLEIL**), and I12 (**DIAMOND**). The potential of our new equipment for *in situ* synchrotron experiments will be illustrated by preliminary results recently obtained from these facilities on many scientific cases: direct visualisation and quantification of melt migration at extreme P-T-Stress conditions [2], XRD-CT of C_{60} sample [3], determination of the density of amorphous materials at extreme conditions [4], anelasticity and attenuation in olivine at upper mantle conditions and seismic frequencies [5], etc.
To conclude, we will present the new scientific opportunities our portable device allows for studies of phase transition, density, crystallization and deformation under extreme PT conditions.

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High-pressure Pair Distribution Function (PDF) Measurement in the Diamond Anvil Cell using High-energy Focused X-ray Beam

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It has been shown that the atomic pair distribution function (PDF) obtained by high-energy X-ray or neutron diffraction is a powerful tool for studying crystalline, disordered and nano-materials [1-7]. Although the pair distribution function, *G(r)*, is simply another representation of the diffraction data, real space exploration of the data has advantages especially in the case of materials with significant structural disorder [2,4]. The total scattering, including Bragg peaks as well as diffuse scattering, contributes to the PDF, and is particularly useful for characterizing aperiodic distortions in crystals [4]. Because of the potentially important role of liquids and disordered solids in the Earth, it is promising for Earth sciences to use the PDF method to characterize the structural variation in short, intermediate and long range orders under extreme conditions of high pressure and temperature [7-9].

However, acquiring PDFs using a large unfocused beam for high-pressure diamond anvil cell (DAC) experiments results in low intensity imposed by small sample chamber of the DAC, long time data collection, parasitic scattering from the upstream slits, diamonds and gasket background contributions, making PDF analysis at pressures much higher than a pressure of 10 GPa not yet possible. Existing high-pressure PDF studies have been limited to low pressures (<10 GPa) [7,8]. Recently, we have succeeded in focusing hard X-ray beam (80 keV) down to a size of 15 μm [10]. Here, we present our recent progress of pair distribution function (PDF) determination under extreme conditions using diamond anvil cell and the high energy X-ray focusing at the beamline X17B3, the National Synchrotron Light Source, Brookhaven National Laboratory. Some examples of the compressibility properties of nanoparticles, such as n-Au and n-Ag, in the diamond anvil cell under quasi-hydrostatic conditions at pressures up to 50—70 GPa will be presented.

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The Nature of the Fe-graphene Interface at the Nanometer Level [1]

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The emerging fields of graphene-based magnetic and spintronic devices require a deep understanding of the interface between graphene and ferromagnetic metals. In this work a detailed investigation at the nanometer level of the Fe-graphene interface was carried out by combining several synchrotron radiation techniques: angle-resolved photoemission from valence band (Figure 2,3), high-resolution photoemission from core levels (Figure 1), near edge x-ray absorption fine structure and x-ray magnetic circular dichroism. The body of experimental data was rationalized by spin polarized density functional theory calculations. We demonstrated that the iron film can be either deposited on top of or intercalated beneath graphene forming heterostructures with different electronic and magnetic properties. Notably, calculations and experimental results show that iron can strongly modify the graphene band structure by lifting the π band degeneracy (Figure 3): the majority spin band maintains its linear dispersion close to the Fermi Level, whereas the minority spin band strongly hybridizes with Fe 3*d* levels and remains pinned at about 2.5 eV. The formation of a spin polarized massless Dirac fermions band as a consequence of the interaction with a ferromagnetic layer demonstrates that graphene can be used for the realisation of spintronics devices.

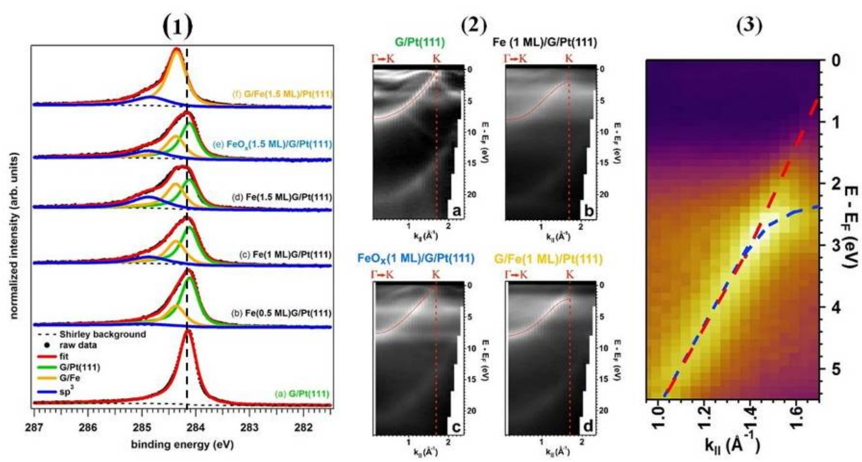

Figure (1) C 1s photoemission line (hv=550 eV) after separation into into single chemical shift components for (a) G/Pt(111) system (b,c,d) after Fe deposition, (e) oxidation and (f) intercalation at 600 K. (2) micro-ARPES acquisition (hv=74 eV) in the Γ to K direction of G for (a) G/Pt(111) system (b) after Fe deposition, (c) oxidation and (d) intercalation at 600 K. Dashed red line are for the theoretical position of the G K point in a free-standing layer. (3) Enlarged view of micro-ARPES of G/Fe(1 ML)/Pt(111) close to the G K point. Underlined with dashed red and blue lines spin majority and minority G π band, respectively.

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Observation and Control of Novel Quantum Phenomena in Artificial Structures of Strongly Correlated Oxides

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Controlling the electronic states of strongly-correlated oxides using artificial structures has heralded the possibility of creating new multifunctional properties in ways that would not have been possible by using single-phase bulk materials [1]. For designing the functionalities of the artificial oxide structures, the precise determination of their electronic structures is indispensable. In order to investigate the novel quantum phenomena appearing in oxide artificial structures, we have constructed an angle-resolved photoemission spectroscopy (ARPES) system combined with a laser molecular-beam epitaxy (laser MBE) oxide-film growth system. The distinctive feature of this system is the direct connection from the spectrometer to the laser MBE chamber; thin film samples can be transferred quickly into the photoemission chamber without breaking ultrahigh vacuum. The system is installed at a new undulator beamline BL-2A MUSASHI at the Photon Factory as an endstation.

By optimally combining sophisticated oxide growth techniques and advanced analysis techniques using synchrotron radiation, we have designed and controlled the novel quantum well (QW) states emerging in digitally controlled ultrathin films of strongly correlated oxide $SrVO_3$ [2,3]. The observed metallic QW states in $SrVO_3$ ultrathin films exhibit characteristic features reflecting their strongly-correlated nature [2]. The successful fabrication of a metallic QW structure based on a strongly correlated oxide demonstrates the capabilities of the developed "*in-situ* ARPES – laser MBE system" for exploring the fundamental physics and extraordinary functionalities of strongly correlated oxides.

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Investigation of Complex Solutions underShear and Pressure

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The unmatched tribological performance of articulated joints is due to both the properties of the cartilage itself and the assumed self-organization of the molecules in the synovial fluid (SF) and at the surface of cartilage.[1] The components of the SF account for the response of synovial tribological system to different load and shear conditions by re-structuring. Thereby they provide extremely low friction coefficients under low and high pressures up to several tens of MPa and different shear rates.[2,3]

In order to investigate how the different constitutes of the synovial fluid work under high pressure and shear conditions we have developed two sample environments which allow us to investigate their self-assembly behaviour in situ by means of small angle scattering. A microfluidic setup allows for measuring the structure of macromolecules in solution at shear rates of more than 100ks^{-1} with nanofocused x-ray beams. The second environment utilizes a fully working rheometer with pressure option (300bar) to test the effect of shear and pressure on the bulk solution and on interfaces as this rheometer has the option to place wafers inside.

In first experiments we investigated highly concentrated protein solutions of lysozyme, which show a shear thinning behaviour. In the course of the experiments we observed a decrease in the structure factor showing an influence of the shear rate on the protein/protein interaction.

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On the characterization of ultra-precise mirrors for the European XFEL by use of slope measuring deflectometry

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Upcoming facilities like the European XFEL will require long and ultra-flat X-ray mirrors of utmost precision for the beam offset and distribution system [1]. The heat load on these mirrors ranges from several Watts in the time average to many kW during FEL pulse trains on the millisecond time scale. The maximum peak-to-valley figure error allowed is less than 2 nanometers along the entire aperture length of 800mm, to enable a wave front preserving transport of photons along very long distances from its source in the undulator section to the experimental hall. Thus it is mandatory these optics to be inspected by use of metrology devices of comparable precision before and after surface finishing as well as in the mounted state. The topography of these mirrors will be inspected by use of slope measuring deflectometry [2] under face side condition the state of their final alignment. We will show and discuss first results of measurements performed on 950mm long flat-distribution mirrors before the final finishing by use of deterministic surface finishing (EEM in this case) as well as on a prototype of a 950mm long bimorph-mirror.

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An XUV At-Wavelength Metrology Facility at BESSY-II

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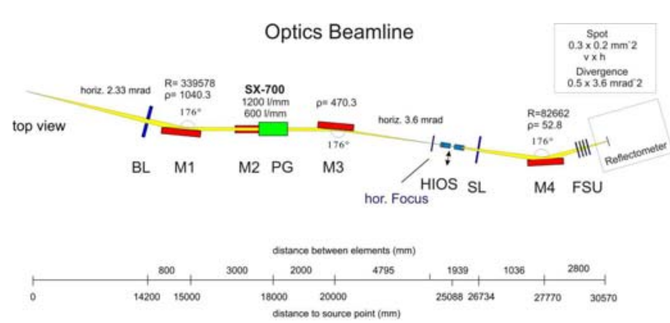
A technology center for production of high precision blazed-gratings was established at BESSY-II in Berlin. Within this project a new Optics Beamline and a versatile Reflectometer has been set up for at-wavelength characterization and calibration of the in-house produced gratings and novel nano-optical devices as well as mirrors, multilayered systems etc.

The collimated Plane Grating Monochromator (c-PGM) beamline is placed at a BESSY-II bending magnet section. According to its purpose, this beamline has specific features, such as: very high spectral purity, provided by two independent high order suppression systems - HiOS (a four-mirror arrangement of different coatings which can be inserted into the beam at different angles) and a filter and slit unit (FSU) (with 12 absorber filters of different thickness and materials), an advanced system for suppression of stray light and scattered radiation and a broad energy range between 10 eV and 2000 eV.

The new 4-circle and 6-axes Reflectometer is able to incorporate real lived-sized gratings. The samples are adjustable within six degrees of freedom by a newly developed UHV-tripod system carrying a load up to 4 kg. The reflectivity can be measured between 0 and 90 degrees incidence angle for both s- and p-polarization geometry. A variety of detectors will provide an extended dynamic range and different angular resolution.

This novel powerful metrology facility has gone into operation recently and is now open for users. First results on optical performance and measurements on multilayer grating and will be presented at the conference.



Financial support of the European Community is gratefully acknowledged (ERDF-contract no. 20072013 2/43)

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VUV Extra-focus Principle and its Application to High Performance Grating Monochromators

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A new monochromator called an extra-focus constant-included-angle varied-line-spacing (VLS) cylindrical-grating monochromator (extra-focus CIA-VCGM, or EF-CIA-VCGM) is described [1]. This monochromator is based on the Hettrick–Underwood scheme where the plane VLS grating is replaced by a cylindrical one in order to zero the defocus at three reference photon energies in the vacuum ultraviolet range. It has a simple mechanical structure and a fixed focus spot with high performance over a wide energy range.

Furthermore, its mechanical compatibility with a standard variable-included-angle (VIA) VLS plane-grating monochromator (PGM) allows convenient extension into the soft-X-ray range. The VIA-PGM/NIM hybrid monochromators have been applied to the beamlines with the photon energy range crossing VUV to soft X-ray for many years. However, some difficulties can hardly be solved, such as low photon flux and not so easy alignment in normal incidence monochromator (NIM) mode. A new design concept is presented in this study that an EF-CIA-VCGM, instead of a NIM, is combined conveniently with a VIA-VPGM. By adopting this new extra-focus dual-mode VLS grating monochromator (EF-DM-VGM), the difficulties above could be solved and a high performance can be achieved with low technical cost.

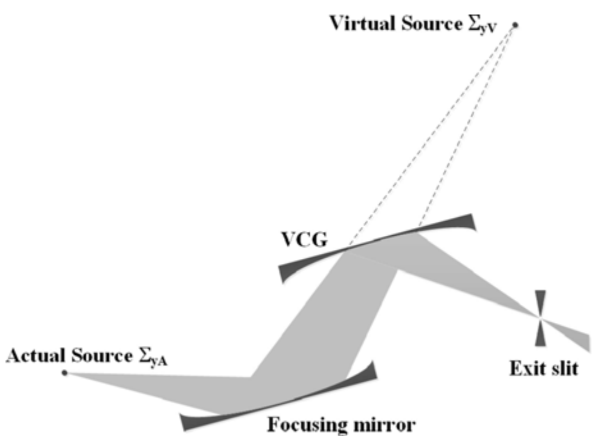

Fig.1 EF-CIA-VCGM schematic diagram

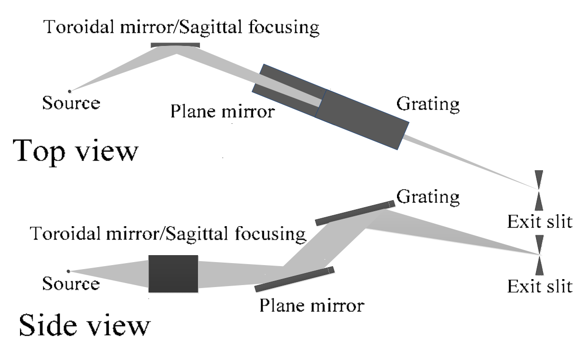

Fig.2 EF-DM-CGM schematic diagram

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X-ray Grating Interferometry for at-Wavelength Wavefront Metrology

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We will present an X-ray grating interferometer setup dedicated to quantitative in-situ, at-wavelength metrology measurements of X-ray optics at the Optics beamline [1] of the Swiss Light Source. The X-ray grating interferometer is based on a diffraction grating which splits the beam into the ±1st diffraction order and creates downstream of the grating a constructive interference pattern at the fractional Talbot distances. An absorption grating installed in front of the detector is used to resolve the interference pattern. From the latter the spatially resolved wavefront can be reconstructed with an accuracy of the order of 10 nrad, allowing to investigate distortions induced by optical elements [2,3]. The realized X-ray grating interferometer is transportable and can be moreover operated on a single-shot basis enabling its use as a diagnostic tool at the SwissFEL. The potential of X-ray grating interferometry at XFELs to investigate the optical elements installed at a beamline and furthermore of the photon source position in the undulator section was already demonstrated [4,5].

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Diffraction Imaging for In-situ Characterization of Double-crystal High-heat-Load X-ray Monochromators

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Development of wavefront preserving high-heat-load x-ray optics becomes increasingly important since coherence properties of x-ray sources are considerably improving. A major effort in the design of double-crystal monochromators is dedicated to minimization of thermal distortion on the first crystal, a dominant factor contributing to wavefront distortion and to instability of the exit beam. Advanced characterization methods have been developed over the years which include measurements of the angular profile of the exit beam using crystal analyzers [1], measurements of the second crystal rocking curve at selected portions of the exit beam [2] and x-ray interferometry [3]. Here we present a new method based on sequential diffraction imaging of the cross section of the exit beam at different angular positions of the second crystal on its rocking curve. The angular profile of the exit beam is extracted from a single rocking curve measurement. Under normal operational conditions of double-crystal high-heat-load monochromators the angular information obtained in the imaging plane can be directly related to thermally induced slope error on the first crystal. The new method can be easily implemented in a variety of beamline arrangements since it requires only an addition of an area detector for imaging of high order reflections (e.g., Si 333). The method was recently used for characterization of a cryogenically cooled double-crystal Si 111 monochromator at 20-ID beamline of the Advanced Photon Source. Preliminary experimental results are reported and compared to results of finite element analysis.

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X-ray Micro-focusing with Off-axis Ellipsoidal Mirror

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At synchrotron radiation facilities, orthogonally-placed two mirrors, so-called the Kirkpatrick-Baez geometry, are commonly used as focusing optics. In this geometry, elliptical-cylinder shape mirrors which focus light in one dimension are typically utilized for the micro-/nano-focusing [1]. Here, mirrors with ellipsoidal shape (Fig. 1), which can produce two-dimensional focus with a mirror, will improve the focusing efficiency and the stability of focusing system. However, difficulties in a manufacturing process prevent a development of ellipsoidal focusing mirrors with a radius of curvature of a few millimeters in the short axis direction in the hard-x-ray region.

Our group has been aiming at the realization of nano-focusing off-axis ellipsoidal mirrors. We have developed a surface profiler and surface processing machines for ellipsoidal mirrors. Surface profiler is based on a precision stitching interferometer. Surface processing machines are based on a precision polishing. These were developed for the improvement of surface roughness and the surface figure correction of ellipsoidal mirrors [2]. Using these techniques, we fabricated an ellipsoidal focusing mirror with surface roughness of 0.2 nm (root-mean-square) and surface figure error height of ~10 nm. As a result of evaluations of the mirror at BL29XUL of SPring-8, we obtained focusing beam size of 0.5 μm (in the meridional plane) (Fig. 2) × 1.2 μm (in the sagittal plane) at full width at half maximum at an x-ray energy of 7 keV using the knife-edge scanning method with a gold wire. The developed fabrication techniques are expected to be applied for the fabrication of nano-focusing ellipsoidal mirrors.

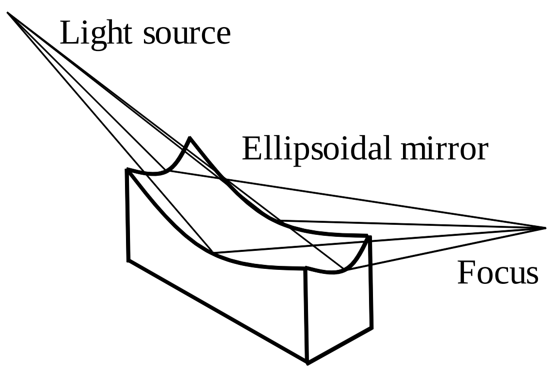

Figure 1. Schematic of off-axis ellipsoidal focusing mirror

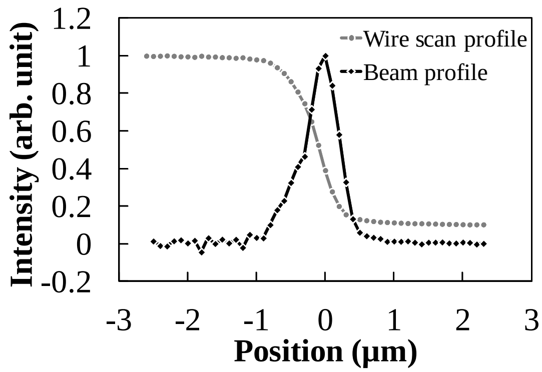

Figure 2. Observed focusing beam profile

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A Spectroscopic 3D Tomographic Investigation of Structure, Morphology and Interface Properties in Sintered Nano-Silver Die-Attach Layers.

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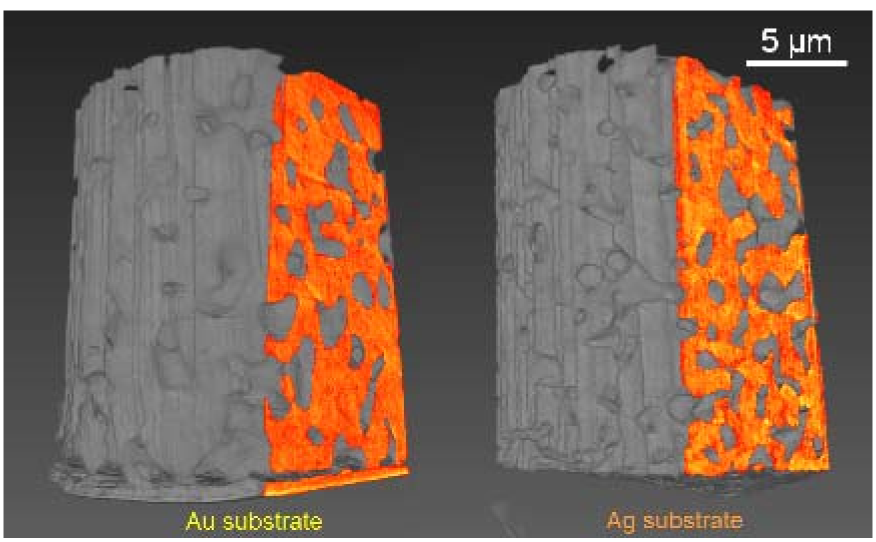
High-power electrical components found in automotive, renewable energy and high-performance electronic applications require reliable, thermally and electrically rugged power modules. These demands must be met by using state-of-the-art environmentally friendly packaging technologies, with one of the most promising approaches based on using sinterable metals instead of conventional solder. The electronic devices usually assembled by stacking a direct-copper-bonded (DCB) substrate (with Cu, Ag or Au coating) and a chip, with sinterable metal in between.

We used non-destructive hard x-ray transmission microscopy (TXM) to obtain 3D tomographic images of the microstructure of the sintered Ag in bulk and at different interfaces: sintered Ag with Cu, Ag and Au on DCB substrate. It was discovered that the substrate material had significant impact on the silver's morphology, with a pronounced depletion layer only a few nanometers thick forming near the interface with Au-coated substrate. Significant changes were also observed upon accelerated aging at the interface between nanosilver and Cu-coated substrate. These findings have important ramifications for the development of next-gen, high-reliability electronic power devices.



High-Throughput and Automated System for SAXS/USAXS Experiment for Industrial Use at BL19B2 in SPring-8

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A highly automated system combining a sample-transferring robot with focused SR beam has been established for small-angle and ultra small-angle x-ray scattering (SAXS/USAXS) measurement at BL19B2 Engineering Science Research I, a bending magnet beamline of SPring-8. High-throughput data collection system can be realized by means of x-ray beam of high photon flux density concentrated by a cylindrical mirror, and a two-dimensional pixel detector PILATUS-2M. For SAXS measurement with the x-ray energy of 15–30 keV, we can obtain high-quality data within 1 minute for one exposure using this system. The sample-transferring robot has a capacity of 90 samples with a large variety of shapes, and can automatically control the irradiated position on the sample. The experimental environment of SAXS/USAXS can be switched easily and automatically. It is notable that the robot has been designed as portable in order to vary the camera length from the sample to the detector fixed at the downstream end of the beamline. This system enables us to observe x-ray scattering covering a wide q-range of 0.005–3 nm^{-1} with the camera length of 0.7–42 m. The fusion of high-throughput and robotic system has enhanced the usability of SAXS/USAX capability for industrial application. We have launched the SAXS/USAXS measurement service on demand at a minimum beamtime of 2 hours.

Industrial Research on Catalysis at Diamond Light Source

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The industrial user programme at Diamond is continuously growing with over 80 companies now making use of beamline and offline facilities. The Industrial Liaison Office (ILO) started its operation in 2007 in order to support all the existing and new customers interested to carry out experiments in various applications. One of the key roles for the ILO team is to gain real understanding of the customers' needs and find the best ways to solve real industrial problems by applying synchrotron-based methodologies.

The team of six scientists covers a wide range of expertise, covering macromolecular crystallography, X-ray absorption spectroscopy, small-angle X-ray scattering, X-ray powder diffraction and small molecule crystallography. The myriad of ways in which we support industrial scientists include running a mail-in service across a number of techniques, providing a full analytical service where we translate research problems into solutions, and collaborating on large research projects.

The requirements for high selectivity and activity of catalysts are among the most crucial demands for a successful commercial application. Therefore, catalyst characterisation provides a unique opportunity for industry to develop new challenging materials for energy, chemistry and environmental technologies. Over the past decades, great efforts have been devoted to developing methods for catalyst characterisation under real operating conditions.

By increasing our understanding of individual industry sector's needs, the Industrial Liaison team at Diamond is open for collaboration with industrial partners in various applications. Specific examples of our collaborative work with industrial partners on catalysis research will be highlighted during my presentation.

In Operando Study of the High-Voltage Spinel Cathode Material LiNi0.5Mn1.5O4 using Two Dimensional Full-field Spectroscopic Imaging of Ni and Mn within 40 nm Resolution

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Combined full field Transmission X-ray Microscopy (TXM) and X-ray Absorption Near Edge Structure Spectroscopy (XANES) techniques were implemented at the hard X-ray microscope at ANKA to follow in operando the chemical phase transformation as well as the microstructural evolution of cathode materials upon operation within an electrochemical cell.

LiNi0.5Mn1.5O4 spinel cathode was studied during the first discharge cycle. The spatial distribution and electrochemical process of the spinel material with spherical granules of 30 µm and 3 µm crystallite size was investigated. The spectroscopic imaging of the cathode within field of view of 40x32 µm² and spatial resolution of 40 nm has revealed an increase of the LiNi0.5Mn1.5O4 granule size during lithiation providing an insight into the effect of the particle size and morphology on the electrochemical process. The chemical elemental distribution and the content of the different oxidation states of the two absorbing elements (Ni and Mn) have been determined in operando from the XANES imaging. A gradual increase in the content of the oxidation state Mn3+ from 8 % up to 64 % has been recorded during the discharge from 5 V to 2.7 V. The study of the local oxidation reduction behavior of Mn3+ reveals a reversibility aspect in the local electrochemical reaction of Mn4+ toward Mn3+ in areas located in the center of the aggregate as well as in areas closed to the electrolyte. During the discharge process, a mixture of Mn3+ and Mn4+ has been detected while only single electron valence states have been found in the case of Ni. Probing the chemical changes during the discharge using two-dimensional XANES reveals spatial differences in the electrochemical activities of the two absorbing elements Ni and Mn.

I12: The Joint Engineering, Environment & Processing (JEEP) Beamline at Diamond Light Source

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I12 is the Joint Engineering, Environmental and Processing (JEEP) beamline, constructed during Phase II of the Diamond Light Source [1]. I12 is located on a short (5m) straight section of the Diamond storage ring and uses a 4.2 T superconducting wiggler to provide polychromatic and monochromatic X-rays in the energy range 50-150 keV (Fig. 1). The beam energy enables good penetration through large or dense samples, combined with a large beam size (1 mrad hor. × 0.3 mrad ver.) The beam characteristics permit the study of materials and processes inside environmental chambers without unacceptable attenuation of the beam and without the need to use sample sizes which are atypically small for the process under study. X-ray techniques available to users are radiography, tomography, energy-dispersive diffraction, monochromatic and white beam 2D diffraction/scattering and small-angle X-ray scattering (SAXS). Since commencing operations in November 2009, I12 has established a broad user community in materials science and processing, chemical processing, biomedical engineering, civil engineering, environmental science, palaeontology and physics.

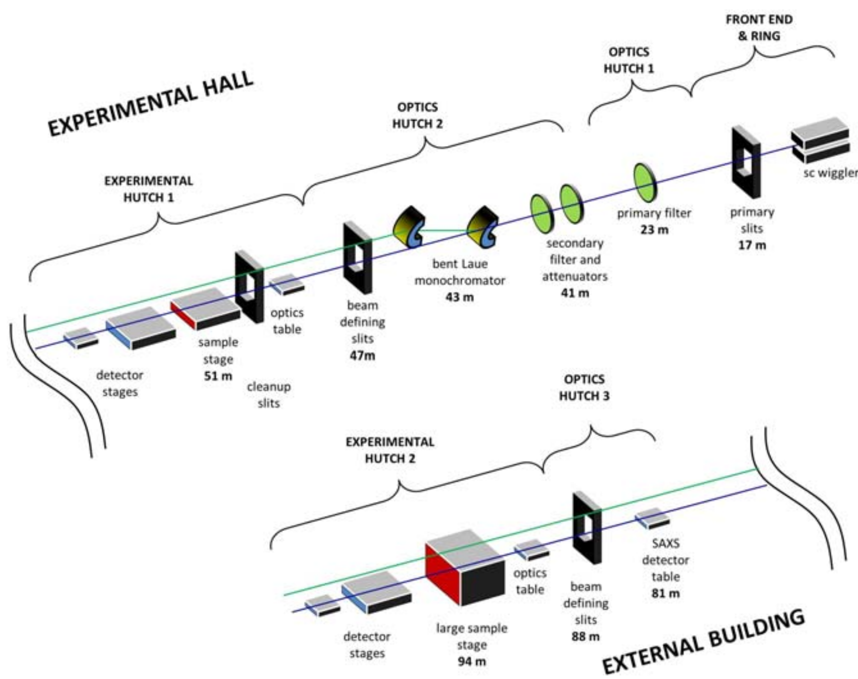

Figure 1: Schematic optical and functional layout of the I12 JEEP beamline.

References

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High Temperature X-Ray Micro-Tomography

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There is increasing demand for 3D micro-scale time-resolved imaging of samples in realistic—and in many cases extreme—environments. The data is used to validate and refine computational models, which are in turn used to more rapidly develop materials with improved performance than could be done by experimental iteration alone. We present work carried out on the x-ray micro-tomography beamline 8.3.2 at the Advanced Light Source. This has included imaging the failure mechanisms in ceramic matrix composite materials while under load at temperatures of up to 1750C (Fig. 1) [1]; pyrolysis of ceramic composite and imaging the degradation of spacecraft heat shields while simulating planetary re-entry conditions. We will describe the instrumentation developed for high temperature tomography, and future directions for coupling 3D imaging with computational modeling for the development of new materials.

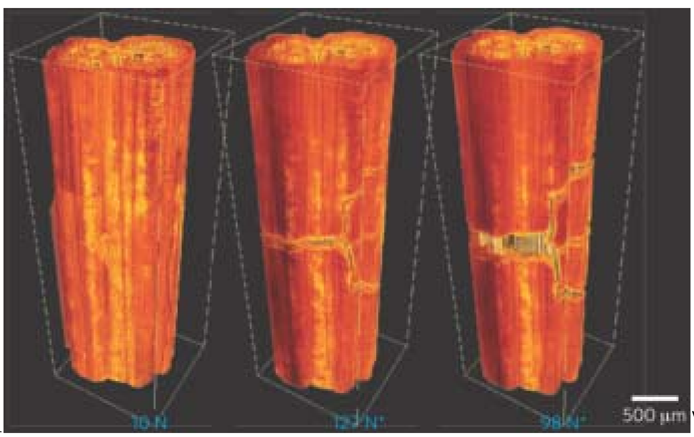
1  \

Figure 1. Time sequence of the failure of a single tow of silicon carbide ceramic matrix composite while under tension at 1750C [1]

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Overview of the Superconducting Undulator Development Program at ANKA

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Superconducting undulators (SCUs) have the potential to reach higher brilliance and flux with respect to the state of the art permanent magnet insertion devices. ANKA is collaborating with the industrial partner Babcock Noell GmbH (BNG) to realize NbTi conduction cooled planar devices for low emittance light sources, and is developing the instrumentation to characterize the magnetic field properties and to measure the beam heat load to a cold bore needed for the cryogenic design of SCUs.

We present here: the most recent results obtained within the ANKA-BNG collaboration, the progress achieved in the development of the instrumentation, and the in house studies on the application of high temperature tape superconductors to the SCU technology.

CHESS Upgrade with Compact Undulator Magnets: Operation Experience and First Results

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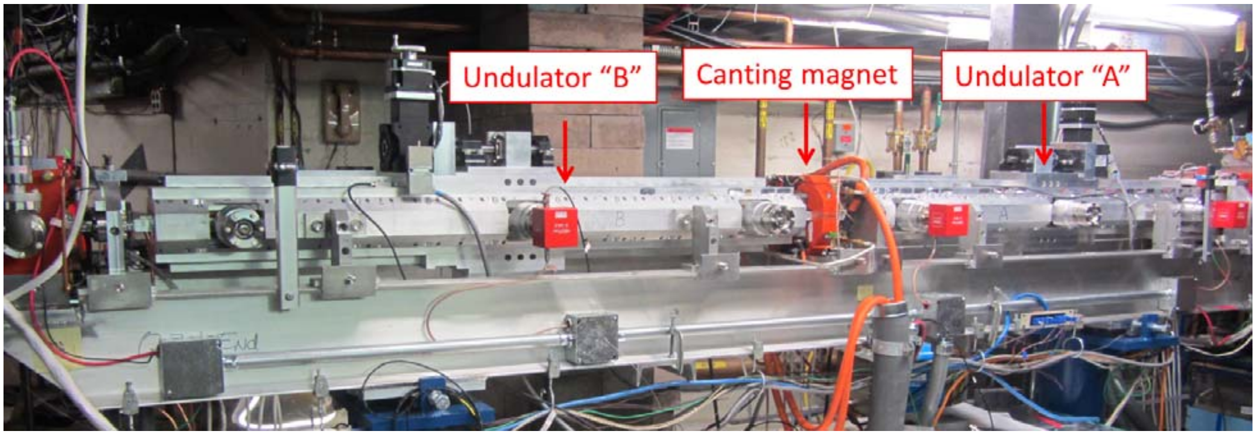
In November 2014 two in-air 1.5m CHESS Compact Undulator (CCU) magnets built by KYMA S.r.l. were installed in Cornell Electron Storage Ring (CESR) in canted arrangement, see picture below, and after few days of commissioning their regular operation started. Presently 5 CHESS experimental stations out of 11 are receiving radiation from these two devices.

CCU magnets are compact, lightweight and cost efficient devices. They have very stable magnetic field integrals independent on "K". This feature greatly simplifies the storage ring operation. The CCU concept was developed at Cornell in 2011 [1] and the test results of the first 1m in-vacuum CCU magnet were presented in [2].

This paper will present the CCU magnet concept and some details of the design and construction. It will also describe the layout of CCUs installation in CESR, their performance and measured characteristics of the undulator radiation. The current status of operation and future plans will be discussed as well.



Two canting 1.5m CCU magnets installed in CESR, 11/5/2014

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Latest Experiences and Future Plans on NSLS-II Insertion Devices

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National Synchrotron Light Source-II (NSLS-II) project will completed in June 2015 and the 3GeV storage ring. Commissioning of the insertion devices (IDs) for the six project beamlines were accomplished smoothly. After closing the gaps of the damping wigglers (DWs), the horizontal beam emittance of 1nm.rad has been achieved.

The composition of the IDs are as follows: There are six 3.4m long DWs with 100mm period-length and 1.8T field at 15mm fixed gap; two 2m long elliptically polarizing undulators (EPUs) with 49mm-period length with 11.5mm minimum gap; two 3.0m long in-vacuum undulators (IVUs) with a minimum vertical aperture (MVA) of 5.0mm; one canted 1.5m long IVU with a MVA of 6.2mm; and one 3.0m-IVU for a long straight-section with a MVA of 5.5mm. Two more identical 1.5m long IVUs and a 2.8m long IVU will be installed in May 2015.

The state-of-the-art "ID magnetic measurement facility" has been constructed to ensure that the field quality of each ID will be acceptable to the NSLS-II storage ring. Our IVUs have unique features including a large rectangular side-window, which allows precise magnetic measurements after vacuum conditioning has been completed at the vendor's facility. The importance of final measurement and fine adjustment is reinforced by the fact that the vendor's pre-baking measurement data could not be reproduced after the final baking and oversea transport.

In this paper, our experience on development, magnetic measurement, installation, survey and commissioning with electron beams will be delineated as well as some future plans.

Experimental Evidences of Light's Orbital Angular Momentum Carried by Helical Undulator Radiation Harmonics

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The phenomenon of higher harmonic radiation from a helical undulator carrying orbital angular momentum (OAM)[1] attracts a great deal of attention because this novel property may be used as a new probe for synchrotron radiation science[2] that would be performed in the next generation diffraction limited light source facility such as NSLS-II and MAX IV.

Although a diffraction limited x-ray source does not yet exist, the 750 MeV UVSOR-III is already a diffraction limited light source in the UV region. In this ring, a tandem-aligned double-APPLE undulator system similar to that in BESSY-II[3] is installed for FEL and coherent light source experiments. Using this configuration, we observed spiral interference patterns between two different harmonic radiations with a scanning fiber multi-channel spectrometer and a CCD camera placed at the end of BL1U beamline. By these measurements, various interference patterns such as single, double, and triple spirals were observed which concur with the theoretical prediction for every mode in the right or left circular polarization[4].

Figure 1 shows images of interference patterns for the topological charge differences of 1, 2, and 3 captured by a CCD camera.

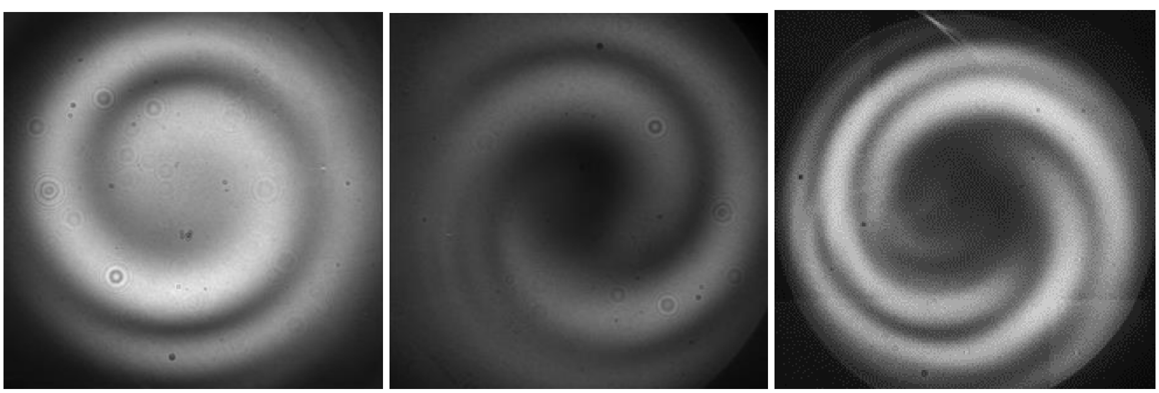

Fig. 1: US; l=0 & DS; l=−1 (left), US; l=−1 & DS; l=+1 (center), US; l=+1 & DS; l=−2 (right)

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Production of High Energy Photons with In-vacuum Wigglers

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Small gap wigglers become more and more attractive to produce high photon fluxes in the hard X-ray photon range. They use magnet blocks of high magnetization which resist much better to heating (baking, synchrotron radiation) than in the past, produce high magnetic field with numerous periods and are very compact. They also are a very good alternative to superconducting technology which requires special infrastructure, heavy maintenance and is not running cost free. SOLEIL has designed and built an in-vacuum wiggler of 38 periods of 50 mm producing 2.1 T at a minimum gap of 5.5 mm to delivered photon beam between 20 keV and 50 keV [1]. Already in operation, further improvements are presently in progress to push photons towards higher energy, in particular thanks to the operation at lower gap (4.5 mm). MAX IV and SOLEIL, in the frame of collaboration, are presently building an upgraded version of the existing SOLEIL wiggler with the target to extend the spectral at high energy (above 50 keV) but also at low energy (4 keV) with the same insertion device. The design of the existing magnetic system has been modified to reach 2.4 T at a minimum gap of 4.2 mm and includes taper operation to avoid undulator structure in the radiated spectrum at low energy.

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Decreasing the Emittance using a Multi-period Robinson Wiggler in TPS

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Taiwan Photon Source (TPS) has been commissioned; its minimum emittance in the TPS lattice is 1.6 nm. To make the emittance less than 1 nm in an exit TPS storage ring lattice is important. A feasibility study to decrease the effective emittance of the TPS storage ring using a Robinson wiggler was launched to enhance the photon brilliance. Of four poles in one set (defined as one period) of a traditional Robinson wiggler (TRW), each pole is combined with a dipole and a quadruple field strength; the corresponding product of dipole and quadruple field strengths of each pole should be negative. Instead of a one-period TRW, we developed a permanent-magnet multi-period Robinson wiggler (MRW). The quadruple field of a combined function magnet in the storage ring is generally about a few T/m. According to analysis of the beam dynamics, the effect of decreasing the effective emittance becomes perceptible when the quadruple field exceeds 25 T/m; a large gradient of field strength in the combined-function MRW magnet is hence demanded. In this report, the quadrupole field strength of the MRW magnet can be more than 40 T/m at magnet gap 15 mm. The period length of a MRW magnet is 300 mm. The effective emittance in various lattice modes -- achromatic mode, low-dispersion mode, low-emittance mode -- on applying a MRW magnet was studied also for the straight sections of length 7 m. The practicability of a MRW is discussed with regard to the possibility of decreasing the entire effective beam size and beam divergence in various lattice modes. The brilliance will become enhanced 34 % when two MRW magnets (length 5 m) are operated in the low-emittance mode lattice herein.

Serial Crystallography at Free-Electron Laser and Synchrotron Light Sources

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Serial crystallography provides a new paradigm for protein structure determination that can be used when large well-diffracting crystals are not available [1,2]. It can be thought of as powder diffraction, measured one crystallite at a time. It combines several new technologies, including of course high peak brightness X-ray sources, rapid sample delivery [3], high frame-rate detectors, and new software [4]. Using sufficiently short X-ray free-electron laser pulses it is possible to outrun the effects of radiation damage, allowing room temperature measurements at high resolution with a dose thousands of times higher than usually tolerable [5]. High-resolution room-temperature protein structures have been determined from crystals less than 0.01 μm^3 in volume. With crystals larger than this, room temperature measurements are possible at the synchrotron: dose can be limited by increasing the delivery speed [6,7]. It should be possible to perform measurements at MHz rates on individual synchrotron pulses. The method is especially useful for time-resolved crystallography [8,9]. Irreversible reactions can be studied, synchronised with the short pulses, with new sample being constantly replenished.

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Cryo Soft X-ray Tomography for Elucidating Pathogen-cellular Interactions

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Cryo soft X-ray tomography (cryo-SXT) [1] can give valuable information on complex phenomena such as microbial infections [2, 3]. We present here an example of pathogen-host interaction investigation on Hepatitis C virus (HCV). HCV is a major cause of chronic liver disease with an estimation of 170 million people infected worldwide. We have obtained the first complete cartography of the gradual and dramatic cellular modification caused by the stable subgenomic HCV replicon transfected in cell culture at different steps of the viral life cycle [4]. The morphology of the membranous HCV factory web is a cytoplasmic accumulation of large and small heterogeneous vesicles, mitochondria and lipid drops (see figure). In addition, we have investigated the recovery at the cellular level of HCV replicating cells treated with specific antiviral drugs.

The structural study of the viral factories in whole cells provides a powerful tool for the analysis of host-virus interactions and represents a potential platform for the trial of new antiviral drugs and vaccines.

In addition, a comparison between spatial resolution achieved with single and dual axis tomography will be presented.

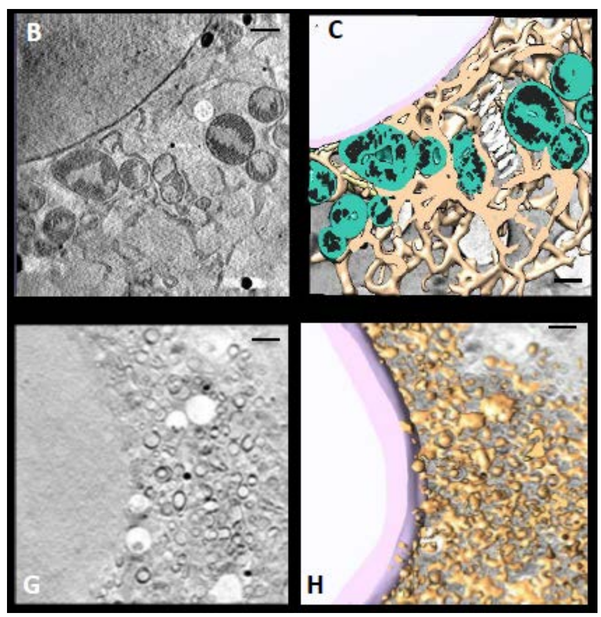

Fig. Cryo-SXT virtual slices of a hepatocyte control cell (B&C) and HCV replicating cell (G&H). Mitochondria have been segmented in blue, ER in yellow. The HCV replicating cell shows a dramatic modification of the cytoplasm. Scale bar: 1µm.

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The New Nanoprobe for Hard X-rays

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The Hard X-ray Nanoprobe (HXN) at the NSLS-II has the world's most ambitious goal for imaging resolution and detection sensitivity. Our aim is to image a sample's morphology, elemental distribution, crystalline ordering, and chemical states with an initial spatial resolution of 10 nm, with the ultimate goal of ~1 nm. Our intensive R&D for multilayer Laue lenses [1] and nanopositioning [2] resulted in the capability to focus hard X-rays down to 11 nm [3] and perform scanning X-ray microscopy experiments with a positioning stability of ~2 nm/h [4]. We undertook substantial effort to create a suitable environment for ultra-high-resolution microscopy experiments by isolating ambient vibrations, and maintaining adequate temperature-stability. Innovative approaches also were implemented in designing the beamline to realize excellent x-ray beam stability. This new hard X-ray nanoprobe is now under commissioning and producing exciting results. The HXN team achieved scanning microscopy capability for fluorescence and differential phase contrast [5] with ~15 x 15 nm resolution. Our presentation will cover the key design features of the nanoprobe, its measured performances, and near-term development plans.

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Wed

Parallel

10:45 AM | Advances in X-ray Detectors I | Wed-A

Detector Developments at DESY for Free-Electron Lasers

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In order to exploit the full potential of the Free-Electron Lasers, DESY embarked on a program to develop new X-ray detectors. A brief overview of the ongoing developments will be given, and first results presented.

For Free Electron Lasers the number of scattered photons per bunch is enough to record a full image, meaning that many photons arrive at the same time, prohibiting photon counting, and leaving integrating detectors as the only alternative. At the same time single photon sensitivity is required. An additional challenge at the European FEL stems from its highly non-uniform time structures with 10 bunch trains per second each containing 2700 bunches (at 4.5 MHz). Finally a very high dynamic range is required for certain imaging experiments, requiring special gain designs. To meet these challenges the AGIPD-system has been under development. Full modules have now been produced and tested at Synchrotron Storage rings. These results will be presented.

An important part of the science performed at Free-Electron Lasers will be in the soft X-ray range, between 250 eV and 3 keV. This energy range is particularly hard for detectors when signal-to-noise and quantum efficiency are of crucial importance. PERCIVAL is a dedicated project to develop a CMOS based imager with up to 13 Mpixels, with 120 Hz frame-rate and having single photon sensitivity down to 250 eV. First back-thinned prototypes have been produced and extensively tested at various synchrotrons. Also these results will be discussed.

Finally, future directions will be briefly presented.

The ePix100 Camera: Use and Applications at LCLS

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The ePix100 x-ray camera is a new system designed and built at SLAC for experiments at the Linac Coherent Light Source (LCLS) [1]. The camera is the first member of a family of detectors built around the same hardware and software platform, where a variety of front-end chips can be integrated. With a readout speed of 120 Hz (matching the LCLS repetition rate), a noise lower than 80 e^- rms and pixels of 50 µm x 50 µm, this camera offers a viable alternative to fast readout, direct conversion, scientific CCD, in imaging mode. The detector, designed for applications such as X-ray Photon Correlation Spectroscopy (XPCS) and with wavelength dispersive spectrometers in the energy range between 2 and 10 keV, comprises up to 0.5 Mpixels in a very compact form factor. In this paper, we report the performance of the camera during its first use at LCLS. As an example in Fig. 1 is shown the histogram of the response of the detector to 8.34 keV photons scattered from silica nanospheres at the XCS instrument [2].

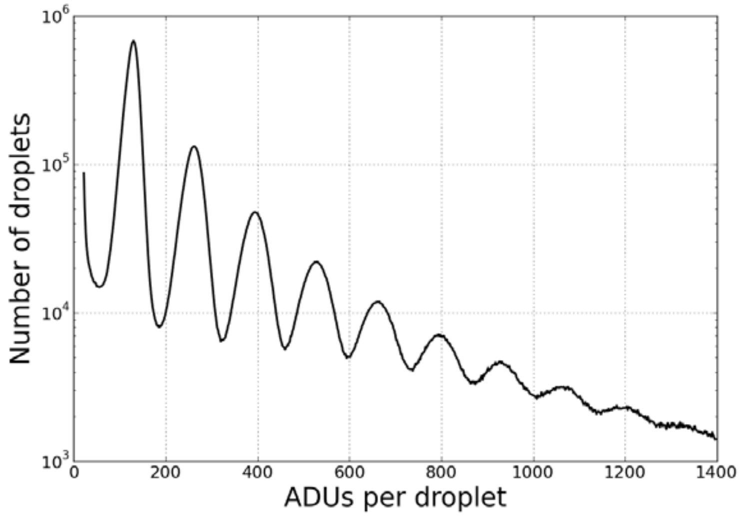

Fig. 1. Histogram of recombined droplets from the speckle pattern generated by silica nanospheres under small angle scattering geometry at the XCS instrument. The single photon peak of 8.34 keV can be seen at ~130 ADU; up to 10 photons can be clearly distinguished.

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Wed

Parallel

11:25 AM | *Advances in X-ray Detectors I* | Wed-A

The High Dynamic Range Pixel Array Detector (HDR-PAD): Concept and Design

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Experiments at synchrotron radiation light sources as well as at next-generation light sources increasingly require detectors capable of high dynamic range operation, combining low-noise detection of single photons with large pixel well depth. XFEL sources in particular provide pulse intensities sufficiently high that a purely photon-counting approach is impractical. Several efforts are underway to produce detectors that address the needs of XFEL users, typically measuring up to 10^3-10^4 photons per pulse [1, 2, 3, 4]. The High-Dynamic Range Pixel Array Detector (HDR-PAD) aims to extend the dynamic range further, with the ultimate goal of measuring up to 10^6 photons/pixel in a single XFEL pulse while maintaining the ability to tolerate a sustained flux of 10^{11} ph/pixel/s at a synchrotron source. Achieving these goals involves the development of fast pixel front-end electronics as well as, in the XFEL case, leveraging the delayed charge collection due to the plasma cloud effect in the sensor [5]. The HDR-PAD builds on the design of the MM-PAD [6], which has a well depth of $4\text{x}10^7$ photons and can tolerate a continuous flux of up to 10^8 ph/pixel/s, but due to the limited speed of its processing circuitry cannot tolerate the instantaneous flux of an XFEL pulse. A first prototype of essential electronic components of the HDR-PAD readout ASIC, exploring different options for the pixel front-end, has been completed and will be fabricated and tested in the coming months. Here, the HDR-PAD concept and preliminary design will be described.

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X-ray Fluorescence Imaging with Energy Dispersive Imaging Detectors and White Beam Synchrotron Radiation

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A novel, "single-shot" fluorescence imaging technique has been demonstrated on the B16 beamline [1] at the Diamond Light Source synchrotron using a white beam bending magnet source with incident X-ray energy up to 45keV illuminating an area of 5x5mm^2. A custom made furnace with 200µm thick metal alloy samples was positioned in the beam with a hole made in the furnace walls to allow the transmitted beam to be imaged with a custom made camera consisting of a 500 µm thick single crystal LYSO scintillator, mirror and lens coupled to an AVT Manta G125B CCD sensor. The samples were positioned 45° to the incident beam to enable simultaneous transmission and fluorescence imaging. The HEXITEC energy dispersive imaging detector [2] was positioned 90° from the sample with a 1mm thick W disk with a 50µm pinhole 13cm from the sample and the detector positioned 2.4m from pinhole. The geometric magnification provided a field of view of 1.1x1.1mm^2 with one of the 80x80 pixels imaging an area equivalent to 13µm^2. The HEXITEC detector had a 1mm thick Schottky-Al CdTe detector connected to the ASIC. The fully spectroscopic ASIC enables every pixel to measure the energy of any detected photon in the range of 3-200keV with an energy resolution of 800eV FWHM at 60keV.

Al-Cu alloys doped with Zr, Ag and Mo were imaged whilst they were heated above the liquid phase transition and subsequently cooled at a controlled rate. The fluorescence images showed that the dopant metals could be simultaneously imaged, with the X-ray flux limiting the fluorescence imaging rate. The elemental distribution could be imaged in tens of seconds and a minute or longer was required to give measurements of the relative concentration of elements in the sample. Figure 1 shows an example of the migration of Ag around the Zr in the sample at different time intervals under isothermal conditions. This technique demonstrated that it is possible to simultaneously image and identify multiple elements without the time consuming need to scan monochromatic energies or raster a focused beam of X-rays. The results from this experiment, plans to improve the experimental configuration and potential applications for this technique and technology will be presented.

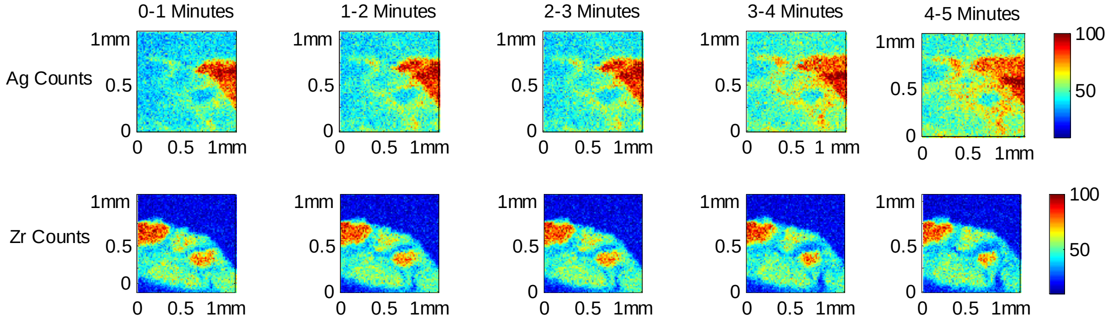

Figure 1. Example of the images of the Ag and Zr elemental distribution at 1 minute time intervals.

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Perspectives in High Resolution, Energy Dispersive and Soft X-ray Imaging using MÖNCH

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MÖNCH is a 25 um pitch charge integrating detector aimed at exploring the limits of current hybrid silicon detector technology. Two fully functional prototypes of size 4x4 mm^2 (25.6k pixels) and 10x10 mm^2 (160k pixels) have been designed and produced in order to test different readout chip architectures [1]. Their perfomance has been characterized in terms of electronic noise, dynamic range and spatial resolution.

The prototypes confirmed a good bump-bonding yield using the in-house process at PSI even for such a challenging pixel pitch, making hybrid detectors competitive with monolithic detectors in imaging applications, with the further advantage of direct conversion even in the hard X-ray range. Additionally, the large signal generated by the absorbed X-rays allows micrometric spatial resolution by interpolating the signal among the pixels collecting the charge cloud produced by each single photon [2]. This makes MÖNCH ideal for applications like inelastic x-ray scattering (IXS), where the detector resolution is currently the limiting factor of the experiment.

An impressive low noise of less than 35 electrons RMS has been achieved using the MÖNCH02 prototype [3]. This energy resolution of better than 300 eV FWHM, together with the capability to simultaneously readout several thousands independent detector elements, makes its usage as high througput as energy dispersive detector feasible in spectroscopic applications. The possibility of simultaneously obtaining high resolution diffraction patterns while detecting the spectrum of the X-rays also opens new perspectives in Laue diffraction and color imaging applications. Soft X-rays down to a few hundreds eV can be detected with single photon sensitivity and a dynamic range up to thousands of photons can be achieved thanks to the dynamic gain switching capability [4].

Preliminary measurements acquired using the current prototypes will be presented and the perspectives opened in photon science by the planned 4x6 cm^2 (1.9M pixels) MÖNCH system will be discussed.

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Superconducting Tunnel Junction X-ray Detectors with Energy Resolution Approaching Statistical Limits

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Superconducting tunnel junction (STJ) X-ray detectors exploit the small ~meV energy gap in superconducting materials to provide more than an order of magnitude higher energy resolution than silicon drift detectors and other semiconductor-based solid state detectors [1]. They can be operated at several 1000 counts/s per detector pixel, and their efficiency can be increased by fabricating detector arrays.

We are developing STJ X-ray detectors based on arrays of superconducting Ta-Al-AlOx-Al-Ta thin films for chemical analysis of dilute samples by fluorescence-detected X-ray absorption spectroscopy (XAS). To avoid the source contribution to the measured linewidth, we have characterized them by direct illumination with the attenuated beam of the grating monochromator (0.2 eV fundamental) at beam line 6.3.1 at the ALS synchrotron. Our STJs have an energy resolution between ~2 and ~5 eV and a peak-to-background ratio >1000 for soft X-rays below 1 keV (Figure 1). The readout electronics currently contributes ~2 eV to the measured linewidth, with the remainder dominated by the statistical fluctuations of the charge generation and tunneling processes [1, 2].

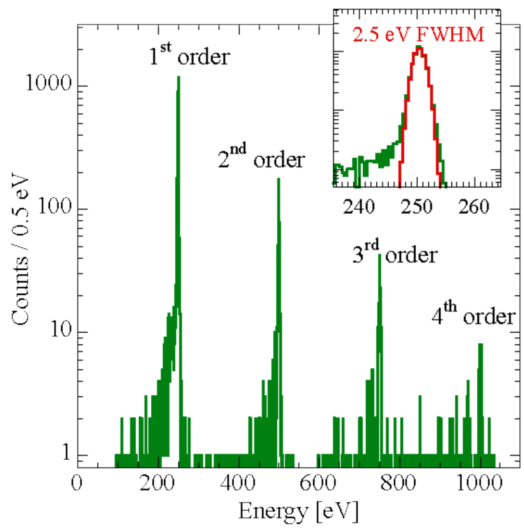

Figure 1: STJ X-ray detector response at ~100 counts/s to the monochromatic beam at ALS BL 6.3.1.

For synchrotron applications, we have built a liquid-cryogen-free cryostat with a cold finger to operate the detectors at temperatures <0.3 K within ~2 cm of a sample at room temperature [3]. A 32-channel preamplifier to read out arrays for improved efficiency is now commercially available [4]. We discuss recent improvements in STJ detector performance, and illustrate their advantages with typical XAS applications.

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Achromatic X-ray Imaging Optics Based on Advanced Kirkpatrick-Baez Mirrors

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Achromatic and high-resolution X-ray imaging optics is highly desirable for X-ray spectromicroscopy. It will allow the production of multicolored X-ray images of small objects. However, conventional X-ray imaging optics, based on a Fresnel zone plate and a compound refractive lens, is affected by chromatic aberration.

To overcome this problem, we have developed an advanced Kirkpatrick-Baez (KB) mirror optics utilizing total reflection [1-4]. It consists of two elliptical mirrors and two hyperbolic mirrors oriented perpendicular to each other (Fig. 1(a)). Moreover, in order to make imaging optics very stable and mirror alignment very easy, recently we have developed monolithic one-dimensional imaging mirrors for advanced KB mirror optics, which have an elliptical shape and a hyperbolic shape on a single substrate (Fig. 1(b)). These mirrors were fabricated with 2-nm figure accuracy using our elastic emission machining (EEM) and stitching interferometers. Performance tests at a demagnification imaging system were first performed at SPring-8. Consequently, ~50-nm resolution was achieved at an X-ray energy of 11.5 keV [2,3]. Then, a magnification imaging test was performed. Figure 2 shows the obtained bright field image of a Siemens star chart, which has features with dimensions as small as 50 nm at the innermost region. A 100-nm feature could be clearly identified and a 75-nm feature is barely visible in the vertical direction. Further, images were taken with different incident X-ray energies to display achromatism. Imaging was possible without losing image quality over a wide energy range of 8–11 keV.

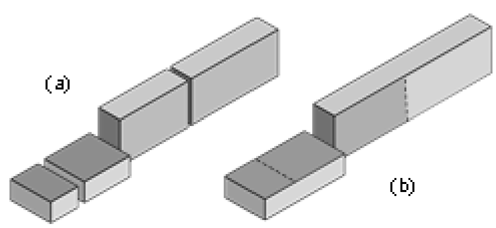

Fig. 1 Advanced KB mirror optics. (a) Separation mirror type and (b) monolithic mirror type.

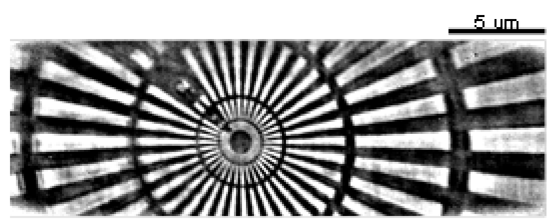

Fig. 2 Bright field image of a Siemens star chart at an X-ray energy of 9.882 keV

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X-ray Refractive Optics: A New Transition for Diffraction Limited Synchrotron Sources

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The use of X-ray refractive optics [1] has rapidly expanded and they are now in common use on various beamlines at 15 synchrotrons in 10 countries. This development has intensified after the successful implementation of transfocators - tunable devices based on refractive lenses [2]. Since 2007, a large variety of transfocators and lens changers were designed and installed at different synchrotrons. Depending on applications the transfocators can provide the following beam conditioning functions in the energy range from 3 to 100 (200) keV: condensers with tunable beam size, micro-radian collimators , low-band pass monochromator [2] and high harmonics rejecters [3].

New advanced parameters of the beam provided by the coming diffraction limited synchrotrons with the reduced horizontal emittance will open up a unique opportunity to build up a new concept for the loss-free beam transport and conditioning systems based on in-line refractive optics. Taking an advantage of the substantially reduced horizontal source size and the beam divergence these new systems integrated into the front-end can transfer the photon beam almost without losses from the front-end to any further secondary optical systems (mirrors, crystals, lenses etc.) or directly to the end-stations. Evidently beamlines will benefit from the possibility to include active moveable lens systems in the front-ends. In this regard, development of diamond refractive optics is crucial [4, 5]. The implementation of the lens-based beam transport concept will significantly simplify the layout of majority of the new beamlines. It will also allow a smooth beamlines transition from the present beam parameters to the upgraded ones, avoiding major optics modifications.

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Development of a Multi-lane X-ray Mirror Providing Variable Beam Sizes

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Grazing incidence mirrors are used on most X-ray synchrotron beamlines to focus, collimate or suppress harmonics. Increasingly beamline users are demanding variable beam shapes and sizes. Active mirrors, mechanically bent or bimorphs, can be adjusted out of focus to increase beam size, but unavoidable polishing errors then introduce strong striations into the beam. We developed the first super-polished bimorph mirror [1] which allowed adjustment of the beam size from sub-micron to 100 µm with reduced striations, but bimorphs respond slowly (over several minutes). We have now developed a new concept to vary the beam size and shape rapidly. The surface of an elliptical mirror is divided into a number of laterally separated stripes, each of which is given a longitudinally dependent calculated height profile to shape X-ray beams to a top-hat in the focal plane [2]. Rapid switching of beam sizes in the range 0.5 to 10 µm is achieved by transverse translation of the mirror to a chosen stripe. Two prototypes with three stripes each have been fabricated, one using the deterministic ion-beam figuring technique (at Zeiss, Germany) with Diamond-NOM [3] data used to iteratively improve the surface figure, and the other using the Elastic Emission Machining technique by JTEC (Osaka). Both mirrors have been tested and characterised using at-wavelength metrology on Diamond's B16 Test Beamline. We envisage that such mirrors will be widely applied to rapid beam-size switching on many synchrotron beamlines. The design concepts and results of the characterisation of the two prototype mirrors will be presented.

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Beryllium Lenses as Collecting Optics for X-ray FEL Radiation

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X-ray FEL beamlines are rather long compared with those at synchrotron radiation facilities. For the Linac Coherent Light Source (LCLS) instruments in the far experimental hall, the x-ray beam is transported ~400 m. The divergence of x-ray FEL radiation is small, but increases approximately linearly with wavelength, λ. At the LCLS the focusing elements, Beryllium (Be) lenses or Kirkpatrick-Baez mirrors, are located in the instrument hutches, far from the source. Consequently, the angular acceptance is limited. The beamline transmission especially at intermediate x-ray photon energies, 3 -6 keV, becomes unsatisfactory. Figure 1 shows the transmission of the Be lenses at the LCLS Matter in Extreme Conditions (MEC) instrument.

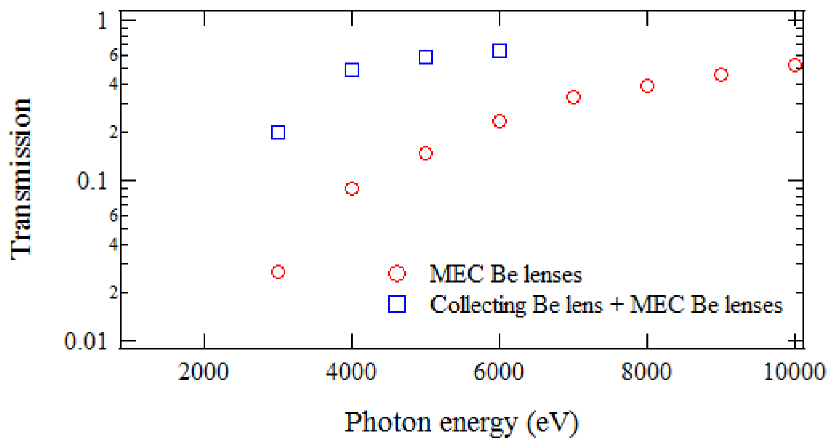

Figure 1. The calculated transmission of the MEC Be lenses in the present configuration and with an additional Be lens acting as collecting optic.

To improve the beamline transmission, it is advantageous to introduce a Be lens at a distance between the x-ray FEL source and the x-ray instrument as a collecting optic. Be lenses are well suited for this purpose. They do not alter the central beam direction, and a single lens set focuses in both the horizontal and vertical directions. This collecting lens can accept the whole x-ray beam and then produce a weakly converging beam. The x-ray beam dimensions are thus matched to the diameter of the Be lenses in the x-ray instrument. Figure 1 shows that the combined transmission of collecting and MEC Be lenses is predicted to improve dramatically. At 5 keV the calculated transmission increases by a factor of 4.

We have installed a manipulator for weakly focusing Be lenses in the x-ray transport tunnel between the LCLS near and far experimental halls. At 5 keV, the transmission of the beryllium lenses with and without the collecting lens has been compared, and the predicted improvement has been confirmed. In addition, the focusing was evaluated by the imprint method.

Phase Preserving Beam Expander for Biomedical X-ray Imaging

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The BioMedical Imaging and Therapy beamlines at the Canadian Light Source are used by many researchers to capture phase-based imaging data. These experiments have so far been limited by a small vertical beam size, requiring vertical scanning of biological samples in order to image their full vertical extent. Previous work has been done to develop a Bent Laue Beam Expanding Monochromator [1] for use at these beamlines, however the first attempts exhibited significant distortion in the diffraction plane, increasing the beam divergence and eliminating the monochromator's usefulness for phase-related imaging techniques. Recent work has been done to more carefully match the polychromatic and geometric focal lengths in a so-called “magic condition” that preserves the divergence of the beam and enables full-field phase-based imaging techniques. The new experimental parameters, namely asymmetry and Bragg angles, were evaluated by analysing knife-edge and in-line phase images to determine the effect on beam divergence in both vertical and horizontal directions, using the beamline's flat Bragg double-crystal monochromator as a baseline. The results show that by using the magic condition, the difference between the two monochromator types is less than 10% in the diffraction plane. Phase fringes visible in test images of a biological sample demonstrate that this difference is small enough to enable in-line phase imaging, despite operating at a sub-optimal energy for the wafer and asymmetry angle that was used.

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X-ray Nanoprobe Project at Taiwan Photon Source

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The hard X-ray nanoprobe facility at Taiwan Photon Source (TPS) provides various X-ray analysis techniques with tens of nanometer resolution, including XRF, XAS, XEOL, projection microscope, CDI, etc. Resulting from the large numerical aperture obtained by utilizing Montel KB mirrors, the beamline with a moderate length 75 meters can conduct similar performance with those beamlines longer than 100 meters. For the beamline design, a horizontal DCM and two stage focusing in horizontal direction is applied. The Montel mirrors are 45 degree cut and placed in a V-shape to eliminate the deformation caused by gravity. This mirror will be made of silica. The slope error of the KB mirror pair is 0.05 μrad which will be accomplished by elastic emission machining (EEM) method. For the endstation, a combination of SEM for quickly positioning the sample, a fly scanning system with laser interferometers, a precise temperature control system, and a load lock transfer system will be implemented.

In this presentation, the design and the construction progress of the beamline and endstation will be reported. The endstation is scheduled to be in commissioning phase in 2016.

OMNY: An Instrument for Tomographic X-ray Nanoimaging

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OMNY is an instrumentation project under way at the Swiss Light Source aiming for 3D nanometric imaging with multi-keV X-rays using ptychographic tomography.

We demonstrate the performance of a prototype-setup that is operating at room temperature and atmospheric pressure at the cSAXS beamline of the Swiss Light Source. The setup provides a stable position between the sample and the beam-defining Fresnel zone plate of better than 10 nm. This position is measured and controlled via dedicated laser interferometry. An unprecedented isotropic 3D resolution of 16 nm was achieved in a tomogram with dimensions of 9x9x3 microns^3. The 3D resolution test object used is based on a nanoporous glass coated with a 37 nm Tantalum oxide layer by atomic layer deposition. The prototype is in routine user operation, we will also present nanoscale measurements of scientifically relevant samples such as catalytic particles.

In order to image radiation sensitive material, such as polymer structures and biological tissue, the final OMNY instrument will operate at cryogenic sample temperatures in ultra-high vacuum and will include a cryogenic sample transfer system. This instrument is currently being assembled and is expected to be operational by mid-2015. We will present design details of this instrument as well as its current status.

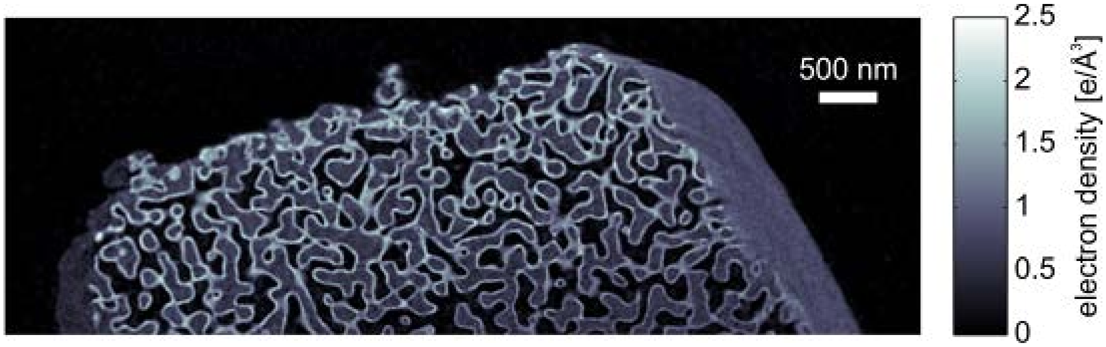

Figure 1: Virtual slice of 10 nm thickness through a 3D tomogram of a nanoporous glass specimen with a 37 nm-thick Ta_2O_5 conformal coating by atomic layer deposition, with an estimated 3D resolution of 16 nm [2].

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X-ray Imaging for *Spatiotemporally* Resolved Studies of Micro-structure Evolution during Technological and Biological Processes

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The talk will focus on developments at ANKA / KIT in the context of current challenges in materials and micro-system technologies and life sciences, where state-of-the-art X-ray imaging techniques provide spatiotemporally resolved information about micro-structure and its evolution during technological and biological processes.

X-ray laminography has been developed

for defect recognition in extended objects [1-4], permitting *in situ* and *in operando* studies from defect generation up to failure. It enables scanning of complete entities with medium resolution, and zooming into region of interests with high resolution [5-8].

An important issue is the development of imaging methods, which enable the visualization of soft tissue, in order to facilitate *in vivo* and *in vitro* investigations, e.g. for developmental biology, functional morphology, nano-toxicology and tissue engineering.

The opaqueness of many organisms impedes *in vivo* investigation by light microscopy. In combination with optical flow algorithms, 4D phase-contrast µCT allows following of spatiotemporal movements, e.g. in order to observe tissues and individual cells during embryonic development [9, 10].

To investigate fast structure dynamics with feature sizes in the micron range and with high temporal resolution, we designed X-ray cine-tomography [15]. The technique enables e.g. new insights into the physiology of small animals by tracking the 4D dynamics of anatomical features as demonstrated by the analysis of screw-and-nut type weevil hip joints [16].

Further development will require an increase in dose-efficiency. Promising routes here include improvement of single-distance phase retrieval at large propagation distances, as well as the use of diffraction based magnifying optics combined with single photon counting detectors [11-14].

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In-situ Liquid Water Visualization in PEM Fuel Cells with High Resolution Synchrotron X-ray Radiography

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The polymer electrolyte membrane fuel cell (PEMFC) is a clean electrochemical energy conversion device considered as a promising powertrain for the next generation of automobiles [1]. PEMFCs use hydrogen and oxygen from air to create electricity with heat and water as the only by-products. A proper management of this water is critical for optimizing the fuel cell performance, reliability and lifetime [2]. However, this challenge can be overcome by improving the structure of the porous material used in the fuel cell.

In this work, we investigated the dominating properties of the porous materials that impact water dynamics in the fuel cell. Visualizations of liquid water in an operating PEMFC was performed at the Canadian Light Source [3], with techniques that have been adapted from our previous work [4-6]. A miniature fuel cell was designed for this experiment, and radiographs were analysed with an in-house image processing algorithm based on the Beer-Lambert law. The X-ray attenuation coefficient of water at 24 keV was measured with a calibration device, and the greyscale values in the raw radiographs were converted into quantities of liquid water thicknesses (cm) [7].

In this communication, we present our methodology for in-situ imaging of liquid water in an operating fuel cell. Our experimental and image processing methodologies facilitate the accurate quantification of fuel cell liquid water content, with a spatial resolution of 10 µm. From this experiment, new insight into the behaviour of in-situ water transport was obtained, and correlations between in-situ liquid water and fuel cell performance will be discussed.

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Novel Technique for Spatially Resolved Imaging of Molecular Bond Orientations using X-ray Birefringence

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Birefringence has been observed in anisotropic materials transmitting linearly polarized X-ray beams tuned close to an absorption edge of a specific element in the material. Synchrotron bending magnets provide X-ray beams of sufficiently high brightness and cross section for spatially resolved measurements of birefringence. The recently developed X-ray Birefringence Imaging technique has been successfully applied for the first time [1] at the Diamond Light Source versatile Test Beamline B16. The orientational order of C–Br bonds of brominated "guest" molecules within crystalline "host" tunnel structures (thiourea or urea inclusion compounds) has been studied using linearly polarized incident X-rays close to the Br K-edge. Imaging of domain structures [1], changes in C–Br bond orientations associated with order-disorder phase transitions [1], and the effects of dynamic averaging of C–Br bond orientations [2] have been demonstrated. The setup uses a vertically deflecting high-resolution double-crystal monochromator upstream from the sample and a horizontally deflecting single-crystal polarization analyser downstream with a Bragg angle as close as possible to 45°. In this way, the rotation angle of the polarization of the beam transmitted through the sample is measured as in polarizing optical microscopy. The theoretical instrumental background calculated from the elliptical polarization of the bending-magnet X-rays, the imperfect polarization discrimination of the analyser, and the correlation between vertical position and photon energy introduced by the monochromator agrees well with experimental observations. The background is calculated analytically because the region of X-ray phase space selected by this setup is not sampled efficiently by standard methods.

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X-ray Beam Induced Current: High Resolution Mapping of Charge Collection Efficiency in Solar Cells

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We present the latest developments in the characterization of thin-film solar cells based on the combination of x-ray beam induced current (XBIC) with x-ray fluorescence measurements (XRF). This is a powerful method to directly correlate electrical properties of solar cells through the charge collection efficiency with elemental distributions.

Based on Monte-Carlo simulations and experimental results from the Advanced Photon Source (beamlines 21-ID-D, 2-ID-D, and 26-ID), we investigate fundamental and practical limitations of XBIC. Comparing XBIC with other techniques – particularly electron beam induced current (EBIC) – we highlight the excellent suitability of high-resolution XBIC for thin-film solar cell characterization.

Applying XBIC and XRF to solar cells with $CuIn_xGa_{1-x}Se_2$ and CdTe absorber layers provides the experimental proof for the excellence of this technique. After thorough data analysis correcting for sample topology and probe-depth effects (self-absorption of fluorescence photons), we demonstrate different examples of XBIC measurements. Figure 1 shows in cross section view that the charge collection efficiency is enhanced at grain boundaries in CdTe solar cells. Figure 2 compares the charge collection efficiency from XBIC measurements to the elemental distribution of copper from XRF measurements. In this case, poor charge collection is observed particularly at grain boundaries, whereas electrically better material is located in the bulk of $CuIn_xGa_{1-x}Se_2$ grains. This is not always the case, but the amount of collected maps provides statistically significant correlation of elemental distribution and charge collection in grain boundaries and in bulk material.

Finally, we present an outlook to experiments that will allow XBIC and XRF *in-situ* and *operando* to enable even further insight into the working principle and limitations of solar cells.

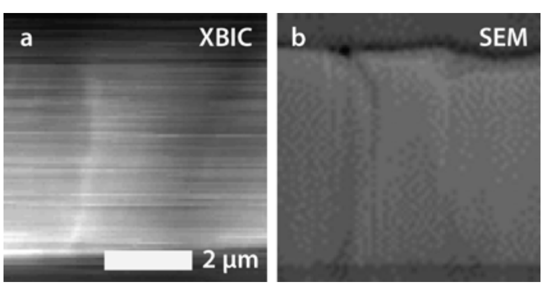

Fig. 1: Enhanced charge collection efficiency measured in cross section by x-ray beam induced current (a) and the same area measured by scanning electron microscopy (b).

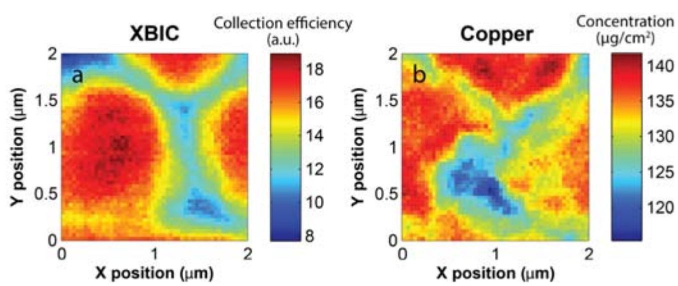

Fig. 2: Plan view of the charge collection efficiency measured by x-ray beam induced current (a) with the copper concentration measured simultaneously by x-ray fluorescence (b).

May the Force be With You: High-speed Atomic Force Microscopes for Synchrotron Sample Holders

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The Surface Science Lab of the ESRF develops since years Scanning Probe Microscopes for X-Rays end-stations [1,2]. The presentation is focused on novel technical developments of custom-made High-Speed AFMs (Figure 1 a,b) for “normal-incidence” and “grazing incidence” *in situ* experiments in X-Ray end-stations. The capabilities of such instruments have been tested at ID03 and ID17 on soft samples (lipids and living cells). The instruments give the possibility to characterize samples exposed to X-Rays at the nano-scale with a speed close to 1 image/second both in air and liquid environment. Therefore, they are suited for studying dynamic morphological events that may be induced or simply complementary observed by X-Rays at nanoscale. This technique aims to identify hierarchical structures of biological, organic and generally soft materials and facilitate the alignment of the X-Ray beam on specific parts of the sample. Moreover, the AFM tip can be employed as nanoindenter for measuring the sample elasticity [3]. As proofs of principle, we shall present quantitatively: 1) the *in-situ* observed change of elasticity of 9L (Figure 1c,d) and F98 living cells upon exposure to an X-Ray dose, 2) the DPPC:DLPC 1:1 bilayers gel-liquid phase transition, observed simultaneously at the mesoscale by *in-situ* AFM and at nanoscale by X-Ray Reflectivity and Surface scattering. Finally, complementary possibilities offered by *offline* AFM measurements to X-Ray scientists in the fields of structural biology and soft matter will be presented.

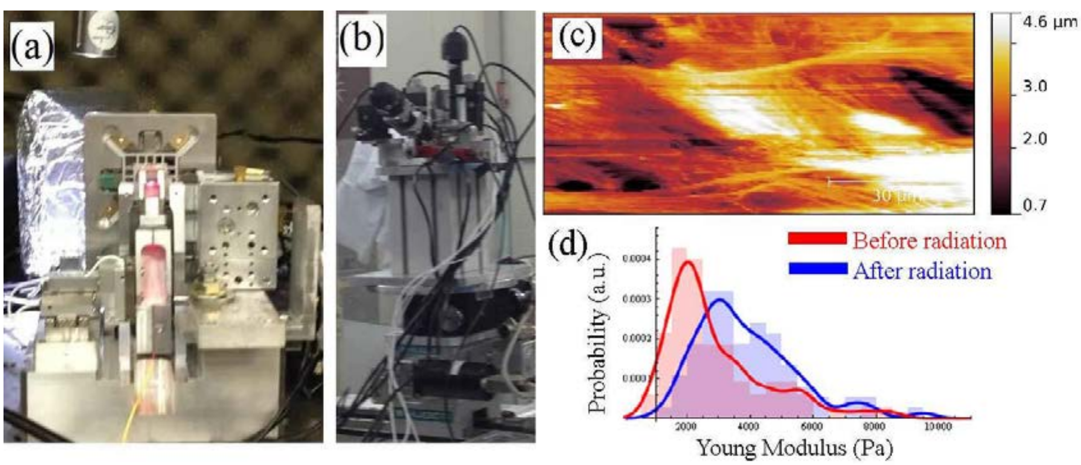

Figure 1: a) HS-X-AFM for ID13 “normal-incidence” experiments; b) HS-X-AFM for “grazing-incidence” experiments mounted at ID17; c) 9L living cells cytoskeleton imaged at ID17 d) Cell elasticity before and after irradiation

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Imaging and CT Modalities at the IMBL of the Australian Synchrotron

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The Imaging and Medical Beamline (IMBL) of the Australian Synchrotron (AS) is now becoming one of the most advanced instruments of this type in the world. It is designed to provide a wide variety of imaging techniques including but not limited to the in-line and analyzed phase contrasts, monochromatic and pink beam imaging. Three beamline's enclosures at various distances, when combined with the 25kW superconducting multipole wiggler and double Laue bent monochromator provide the end user a good choice of beam characteristics ranging from the hi-flux for high resolution and size up to huge 48x5cm beam at 134m from the source with the allowed energy range 17-120kEv. The wide range of the area detectors allows the computed tomography (CT) and tomosynthesis methods to be applied to almost any known X-ray imaging modality. The beamline's data acquisition system is directly linked to the high performance computing facilities tuned for the on-the-fly real-time reconstruction and 3D rendering. Deep integration of the acquisition, reconstruction and rendering facilities allows one to think of the their combination as of a a single system with modular architecture. The system is designed for the fully automated experiments with minimal user interaction. This report summarizes implemented, designed and planned features of the beamline as applied to the imaging experiments. Some latest outcomes of the CT system are presented with the samples coming of different fields of science: Biology, Geology, Paleontology and Medicine [1].

Figure 1: Example of the IMBL CT scan. The sample is a developing flower of the Capitularina involucrata approximately 5x5x10mm in size.

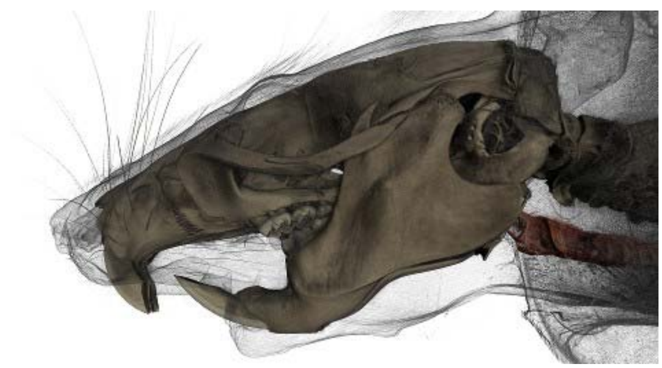

Figure 2: Example of the IBL CT scan using propagation-based phase contrast imaging. The airways of the rat are studied as part of a project focused on the disease associated with Cystic Fibrosis Sample view size: 20x20x80 mm, pixel size ~6micron.

References

[1] More 3D renderings are available publicly: http://www.youtube.com/user/antonmxx

Multiple Energy Synchrotron Biomedical Imaging System

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The continual drive to improve and expand the amount of information extracted from various imaging modalities has led to the use of multiple x-ray photon energies in computed tomography clinical systems [1]. The interest in multiple energy imaging (MEI) is not only with the use of laboratory x-ray source but also with synchrotron x-ray source [2]. A novel MEI system, which prepares a focused polychromatic x-ray, has been developed at the Biomedical Imaging and Therapy (BMIT) bend magnet beamline at the Canadian Light Source. The MEI system is made up of a cylindrically bent Laue single (5,1,1) silicon crystal monochromator and an area detector. Depending on the horizontal beam width of the filtered synchrotron radiation (20 to 50 keV) and the bent radius of the crystal, the size and spectral range of the focused beam prepared vary. For example, using a bent radius of 95 cm and a 50 mm wide beam, a 0.5 mm wide focused beam of spectral range 27 keV to 43 keV was obtained. This spectral range covers the k-edges of iodine (33.17 keV), xenon (34.56 keV), cesium (35.99 keV), and barium (37.44 keV); some of these elements are used as biomedical and clinical contrast agents. With the developed MEI system, a test subject composed of iodine, xenon, cesium, and barium along with water and bone were imaged and their concentrations successfully extracted. The system, its operation, the measurements performed including dose rate to the test subject, and the possible biomedical applications will be discussed.

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Qualifying Biolabel Components for Effective Biosensing by Advanced High-throughput Synchrotron Radiation - SEIRA Methodology

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The need for technological progress in bio-diagnostic assays of high complexity requires both fundamental research and constructing efforts on nano-scaled assay recognition elements that can provide unique selectivity and design-enhanced sensitivity features for reliable high-performance analysis. Indeed, high-throughput capabilities require the simultaneous detection of various analytes combined with appropriate bioassay components. To design bioassays that function as molecular probes, a selective binding event has to be optimized with respect to the over whole physicochemical properties which may influence the successful readout of signals. This constitutes the core of the first design steps to elaborate multi-purposes bioassay platforms.

Nanoparticle induced sensitivity enhancement, and its related application to multiplexed capability Surface-Enhanced InfraRed Absorption (SEIRA) assay formats are fitting well these purposes. SEIRA constitutes an ideal platform to isolate vibrational spectroscopic signatures of bioassay' targeted and active molecules (as antibodies). Accordingly, the potential of diverse targeted biolabels, here fluorophore-labeled antibody conjugates, chemisorbed onto low-cost biocompatible gold nano-aggregates SEIRA substrates has been explored for their use in assay platforms. Extensive areas of dried sample films were analyzed by synchrotron radiation FTIR/SEIRA spectro-microscopy and the resulting complex hyperspectral datasets, containing molecular SEIRA fingerprints, were submitted to automated statistical analysis, namely principal components analysis. Relationships and dependencies between chemical functional groups of the various antibody-fluorophore conjugation systems were determined for revealing their spectral discrimination capabilities.

Consequently, we demonstrate that robust spectral encoding via SEIRA fingerprints opens up new opportunities for a fast, reliable and multiplexed high-end screening not only in biodiagnostics but also in in vitro biochemical imaging.

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Investigating the Microvessel Architecture of the Mouse Brain: An Approach for Measuring, Stitching, and Analyzing 50 Teravoxels of Data

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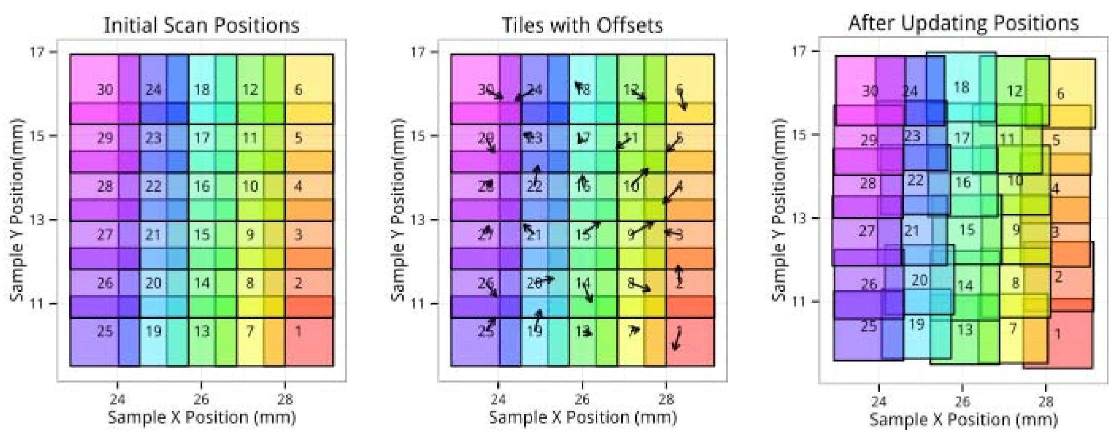
As acquisition speeds and stability improve, new possibilities have been opened in imaging large samples at high resolution. Of particular interest have been massive scale projects like the Human Brain Project and adult Zebra fish imaging. Both involve thousands of measurements at micron resolution to cover mm to cm scales. The task of processing and analyzing such large collections of measurements is exceptionally difficult. In this work, we address the challenge of stitching together terabytes worth of scans in a parallel, distributed manner. Building on the distributed frameworks of Apache Spark and Spark Imaging Layer, we have extended the methods of (S. Preibisch 2009) to work on these images enabling the use of many machines in parallel and drastically accelerating the speed and ease with which these large datasets can be stitched and analyzed.

By automating the acquisition, we conduct all of the scans locally in a zigzag pattern to minimize the effect of motor position drift. Each scan consists of 1000 projections sized 2560 x 2160 (14GVx) with 0.65µm isotropic voxel size. To measure the entire mouse brain approximately 15 steps will be taken in each direction (~3400 total→ 50TVx). A correlation is performed and the maximum value is taken to produce an offset vector. The entire set of positions is then updated in an iterative manner with a smoothing function applied to these vectors.



Each image shows colored tiles and numbers indicating the information covered in a single scan. The images from left to right show the results after acquisition, the new offsets determined by correlation, and the updated positioning from a smoothed version of these offsets

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Enabling Investigations of Liquids and Liquid-solid Interfaces with Soft X-ray Excitation at UHV Conditions

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Capturing biochemical markers by biomolecular films is one of the most promising approaches for the development of highly sensitive and highly selective diagnosis. In particular, future innovative tools for in vitro or point of care diagnostics are expected to rely on this principle.

Analytical techniques which can provide information on coverage, orientation and chemical state of biochemical films are capable of contributing to a purposeful development of such diagnostics. We present a liquid cell, which was designed to facilitate the application of soft X-ray spectrometry for the in-situ analysis of biomolecular films at solid-liquid interfaces. It allows for
- the analysis through a silicon nitride window with a thickness of about 150 nm
- in situ preparation of successive layers by rinsing the window

Currently, after the first successful soft X-ray experiments we are improving the versatility of the liquid cell. Spectrometry in transmission and in various emission geometries will be feasible. Further control devices for the experimental conditions will be added.

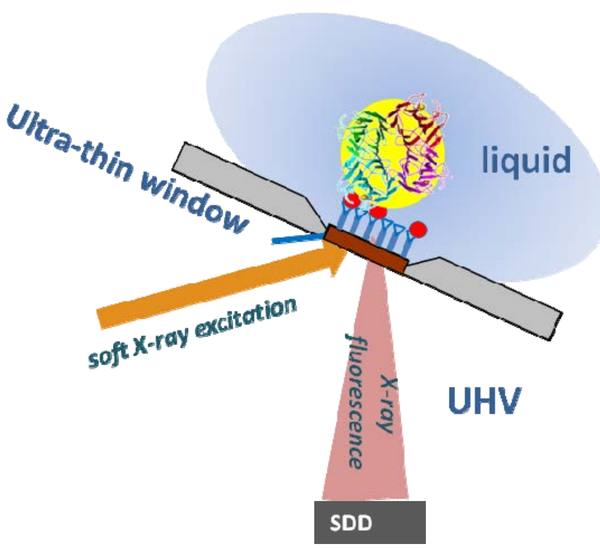

Figure 1: sketch of measurement arrangement [1]

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Development of the XFP Beamline for X-ray Footprinting at NSLS-II

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For over a decade, synchrotron-based footprinting studies at the NSLS X28C beamline have provided unique insights and approaches for examining the solution-state structures of large macromolecular assemblies, membrane proteins, and soluble proteins, for time-resolved studies of macromolecular dynamics, and most recently for *in vivo* studies of RNA-protein complexes. The transition from NSLS to NSLS-II has provided the opportunity to create an upgraded facility for the study of increasingly complex systems; progress on the development of the XFP (X-ray Footprinting for *In Vitro* and *In Vivo* Structural Studies of Biological Macromolecules) beamline at NSLS-II is presented here. The advantages of this beamline will be the high flux density x-ray beam delivered by a focused 3-pole wiggler source in the microsecond to millisecond timescales appropriate for probing macromolecular dynamics while minimizing sample perturbation, and the stable high-current top-off operations mode at NSLS-II, which will maintain the beam at effectively constant optimal integrated flux. The beamline optics and diagnostics enable adaptation of the beam size and shape to accommodate a variety of sample morphologies with accurate measurement of the incident beam, and the upgrades in sample handling and environment control will allow study of highly sensitive or unstable samples. The XFP beamline is expected to enhance relevant flux densities over 40-fold from the standard beam at X28C, allowing static and time-resolved structural analysis of highly complex samples that have previously pushed the boundaries of XF technology. XFP is anticipated to become available for users in 2016.

VIPIC: A Custom-made Detector for X-ray Speckle Measurements

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We have developed a new detector, designed from the ground up to facilitate X-ray Correlation Spectroscopy (XCS) experiments. The device uses state-of-the-art silicon technology [1] to allow continuous, zero dead-time measurements with microsecond resolution. The detector only delivers non-zero data, greatly reducing the communications overhead typical of conventional frame-oriented detectors such as CCDs. The talk will describe how this is achieved and show some preliminary data taken at APS. We will also indicate our plans for future developments along these lines.

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Development of New X-ray Detectors within the Framework of the ESRF Upgrade

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The detector activities at the ESRF in the recent past as well as the plans for future developments have been and are tailored to match the strategic objectives of the ongoing Upgrade Programme of the facility. The priority during the first phase of the ESRF Upgrade has been put on building new enhanced beamlines and equipping them with adapted instrumentation that provides optimum experimental capabilities. In the area of X-ray of detectors this has required for most of the beamlines the development of highly customised instruments built by integrating state-of-the-art detection concepts that in some of the cases involved the combination of various existing advanced detection technologies in the same device. The outcome of this approach will be presented with several illustrative examples.

The second phase of the ESRF Upgrade, started in 2015, aims even further at the future by implementing a new nearly diffraction-limited X-ray source that will boost both the brilliance and the coherence of the ESRF beams. In this context the ESRF has initiated a specific program that addresses the main limitations that will face the existing detectors to fully exploit the new beams. This new program plans the development of complete 2D detector systems as well as to promote and improve several strategic technologies that will be instrumental in construction of the detectors required in the future by the ESRF beamlines. While some of the new technologies and devices will be selected and designed to deal with higher photon fluxes and shorter timescales, an important specific goal of the program is to produce detector systems optimised for the application of coherent scattering techniques, even with high energy photons, in a wide range of scientific areas.

Development of 1D and 2D CdTe Detectors at SPring-8

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We have been developing 1D- and 2D-type high energy X-ray detectors combined with CdTe Schott-ky diode sensors and photon-counting readout ASICs [1-4]. On the CdTe sensor device the front side was deposited with aluminum to form pixelated electrodes and the back side was covered with a single plati-num-electrode. This electrode configuration has the advantage of providing a high Schottky barrier formed on the Al/CdTe interface, and, hence, a benefit to operate the CdTe as an electron-collecting pixe-lated diode.

The 1D-type CdTe sensor assembly was designed as it is compatible with the MYTHEN Si-strip sen-sor. While the silicon strip sensor could directly connect to an ASIC with wires, the CdTe strip sensor couldn't employ the wire-bonding technique because the CdTe crystal easily gave due to mechanical stress during the bonding procedure. In our technical choice the CdTe strip sensor was once bump-bonded to an interposer board with an In/Au-stud bonding method, and, then, the interposer board was wire-bonded to the ASIC. 638 strip-shaped electrodes formed 50 µm in pitch on the CdTe sensor and two sen-sors was adapted on one interposer board with 200 µm dead area in between two sensors. Figure 1 shows the CdTe-strip detector adapted with the MYTHEN MCB.

The 2D-detector module was designed with a pixel pitch of 200 µm × 200 µm and an area of 19 mm × 20 mm or 38.2 mm × 40.2 mm. The SP8-04 ASIC has a preamplifier, a shaper, 3-level window-type discrim-inators and a 24-bits counter in each pixel. The prototype detector with 20 × 50 pixels successfully oper-ated with a photon-counting mode selecting X-ray energy with the window comparator. Excellent energy linearity was achieved between 15 and 120 keV. We performed a feasibility study for white X-ray mi-crobeam experiment with the prototype 2D detector. Laue diffraction patterns were measured during the scan of the irradiated position in the sample. Figure 2 on the left shows a typical Laue diffraction pattern and grain boundary image with a silicon steel sample. In this talk, we will present the specification and performance of CdTe-MYTHEN 1D detector and a large area 2D detector with the SP8-04 ASIC.

Fig. 1 1D CdTe detector

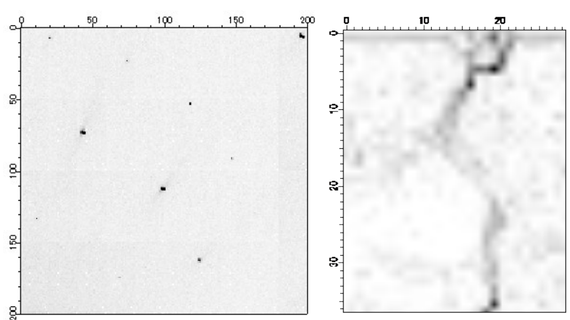

Fig.2 Laue diffraction pattern and grain boundary image with a silicon steel sample

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Large-Area CdTe Pixel Detectors for High-Energy X-ray Diffraction Applications

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A large-area cadmium telluride (CdTe) hybrid photon counting pixel detector suitable for high-energy X-ray diffraction applications at energies from about 8 keV to above 100 keV has been developed. The detector with 300k pixels with a sensitive area of 84 mm × 106 mm and a pixel size of 172 µm × 172 µm is built in hybrid-pixel technology by bumpbonding PILATUS3 CMOS readout ASICs [1] to pixelated CdTe sensors with a large area of 42 mm x 34 mm and a thickness of 1000 µm. Photon counting in each pixel provides noise-free images with no dark signal and at frame rates of up to 500 Hz with readout time of less than 1 ms. In contrast to scintillator-based flatpanels, this direct-converting CdTe detector exhibits a sharp point-spread function, well confined to the pixel size. The detector modules have been thoroughly characterized in terms of quantum efficiency (Fig. 1), point-spread function, long-term stability of CdTe material and count rate capability using an X-ray tube setup and at a synchrotron beamline (BAMline at BESSY II). Moreover first application tests were carried out at several synchrotron beamlines of the European Synchrotron Radiation Facility, Diamond Light Source, and the Advanced Photon Source. The diffraction applications tested with the CdTe detector, including diffraction tomography, powder diffraction, and inelastic scattering, strongly profit from the detector characteristics. The results of characterization and application tests are presented to illustrate the advantages and possibilities enabled by this new detector technology. Following the successful application tests, the development of a large CdTe detector with more than 2M pixels (24 detector modules) was started recently [2].

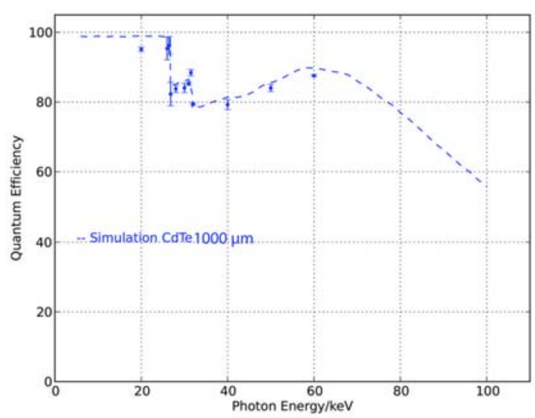

Figure 1: Quantum efficiency of PILATUS3 CdTe module measured at the BAMline at BESSY II.

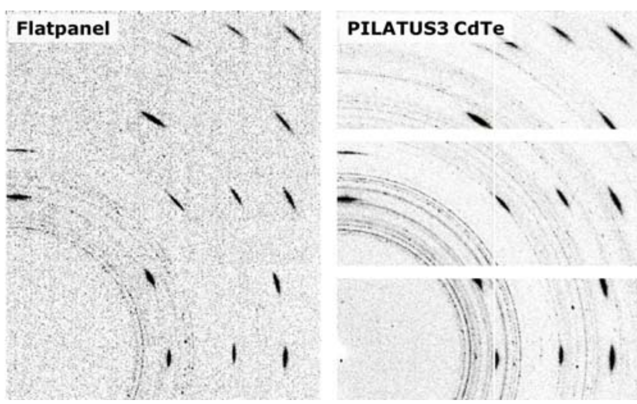

Figure 2: Comparison of data quality of flatpanel and CdTe detector at 46.3 keV. Image courtesy: Marco Di Michiel, High-Energy Scattering Beamline ID15A, ESRF.

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Imaging NEXAFS Detector for Compositional and Structural Analysis

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The Large Area Rapid Imaging Analysis Tool, or LARIAT, provides full-field hyperspectral near-edge X-ray absorption spectroscopy (NEXAFS) imaging through the use of a magnetic projection lens. Using a 1T electromagnet, imaging resolutions of approximately 50 μm can be achieved; while using an 8.5T superconducting magnet, imaging resolution improves by an order of magnitude. The magnetic projection lens additionally ensures rapid imaging, as all electrons emitted from within the imaging area are collected by the detector. Energy filtering is provided by either a grid or electrostatic lenses. The initial LARIAT systems were installed at NSLS and are in the process of installation at NSLS II.

Additional upgrades to the LARIAT systems are being developed, including the application of time-of-flight (ToF) and spot modes. The ToF mode will use high-speed switching electronics to alternate the electrode biases for electron energy filtering. Current modeling suggests energy resolutions approaching 250 meV are attainable. Such an energy resolution would permit the collection of photoemission and Auger data in addition to the unfiltered NEXAFS. Spot mode will permit a user to ‘zoom in’ on interesting features, analyzing both the ‘forest’ and the ‘trees.’ Additionally, combining spot and ToF modes provides the ability to measure an energy spectrum from the radial distribution of electrons in the image spot.

A Wide Dynamic Range X-ray Detector with Silicon-On-Insulator Photon Imaging Array Sensor (SOPHIAS) for SACLA

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Coherent X-ray diffraction imaging (CXDI) experiments with X-ray free electron laser (XFEL) is one of the powerful methods to visualize biological specimen at the nanometer scale. Recently an application to living cells has been reported at the spatial resolution of 28 nm [1]. In order to investigate larger specimen at higher spatial resolution, recording of X-ray scattering from higher intensity to a week signal level of single photon is required. Other XFEL applications also demand X-ray imaging sensor with wider dynamic range with smaller pixels. In order to meet these requirements, we have been developing a detector system with a SOI photon Imaging Array Sensor (SOPHIAS). The sensor design and preliminary production results were reported in Ref. 2. The specifications and evaluation results of the detector system are summarized in Table 1.One of the advantages of this detector is the pixel architecture enabling wide dynamic range. The pixel has high and low gain circuitries with a gain difference of 60 through the division of signal charge [2]. Owing to the charge division, a small pixel size of 30 μm with a peak signal of 10 000 photons at 6 keV has been realized. The evaluation at the SACLA BL3 with XFEL beam was also performed. An X-ray small angle scattering image of cobalt oxide power is shown in Fig. 1. Detailed analysis of the performance together with the system implementation will be reported.

Table 1 Specifications and Performance of the detector for SOPHIAS

Parameters		Value	Units
Sensor	Pixel Size	30	μm
	Pixel Number	1.9 M (891 (V.) x 2157 (H.))	N/A
	Noise[1,2]	0.12	phs. at 6 keV X-ray
	Peak Signal[1,2]	11400	phs. at 6 keV X-ray
	Frame Rate	60	Hz
Camera	Number of sensors	2	N/A
	Pixel Number	3.8 M (2157 (V.) x1782 (H.))	N/A
	Operation Temperature	-25	°C
	Interface	Cameralink full configuration	N/A
	Data Output Rate	7.4	Gbps

1) Results obtained for a sensor. 2) Peak signal is defined as maximum signal satisfying linearity of 3 %

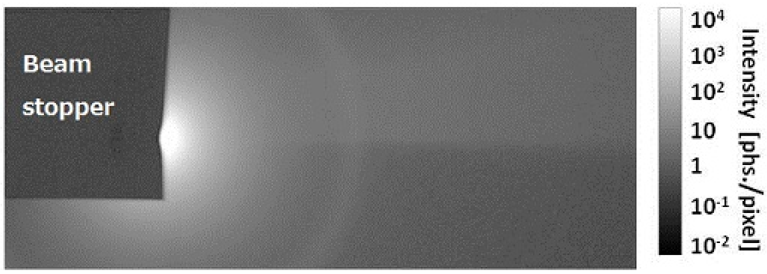

Fig.1 An X-ray scattering image recorded with an XFEL pulse at the photon energy of 6 keV. Small angle scattering with signal level over 10 000 photons was clearly observed around the beam stopper.

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Advances Multilayer Laue Lenses Fabrication

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The demand for high spatial resolution at synchrotron beamlines has pushed the development of nanofocusing optics. At the same time, progress in thin film deposition and nanofabrication techniques broaden the possibilities for fabrication of new devices with larger design flexibility and control. At the crossroads of both fields, multilayer Laue lenses (MLLs) have been receiving increased attention [1]. A MLL consists of a depth-graded multilayer which is subsequently sectioned into a high–aspect ratio structure to become a usable optic. Different types of MLL can be fabricated: flat, tilted, wedged and curved [1]. The types distinguish themselves by either geometry in which the optic is going to be used (flat, tilted) or by the thickness profile of the layers (flat, wedged, curved). In the past few years, focusing resolution using flat MLLs has been demonstrated to approach the diffraction limit [2]. The growing demand for optics that combine high resolution, large working distance, and high efficiency for hard X-ray microscopy is now pushing the development of large aperture and wedged MLLs. In this presentation, we report our progress on the fabrication of large aperture flat and wedged multilayer Laue lenses [3].

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Challenges Toward Single Nanometer Focusing of X-ray Free Electron Laser

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X-ray free-electron laser (XFEL) sources have a peak brilliance 10^9 greater than that of the most powerful third-generation synchrotron radiation sources and can emit high-intensity femtosecond pules with full spatial coherence[1][2] To enhance the characteristics, focusing XFEL is a critical and urgent requirement. We have already established 1μm and sub-50nm focusing of XFEL at SPring-8 angstrom compact free-electron laser (SACLA) [3][4]. Here, multilayer mirror optics for single nanometer focusing of XFEL are discussed in terms of optical design, radiation damage, mirror fabrication, and a wavefront diagnosis to be developed at SACLA. Expected beam size is nearly 5nm and will be used to explore the forefront of nonlinear optics in hard X-ray regime. In this optics, a two-stage Kirkpatrick-Baez (KB) configuration is employed and precision multilayer mirrors with long and steeply curved surfaces are installed to realize a required numerical aperture, to reduce radiation damage, and to have enough working distance. A Pt/C multilayer was tested as a candidate and confirmed to survive under the XFEL irradiation. An at-wavelength wavefront measurement method with a single grating Talbot interferometry is utilized to understand the performance of focusing optics. The measured error in the wavefront will be used for refiguring the mirror substrates. We will present the latest status of the project.

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Focusing with Crossed and Wedged Multilayer Laue Lenses

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Multilayer Laue lenses (MLL) are one of the most promising optics to achieve small focal spot sizes together with high focusing efficiencies for hard x-rays. For very small focal sizes at large numerical apertures and large total apertures it is necessary to use the so called wedged geometry.

We have manufactured a multilayer stack with layer thicknesses according to the zone plate law for a focal length of 6.7 mm at 10.5 keV x-ray photon energy using WSi_2 and Si as absorber and spacer materials. The design with 7000 zones offers a total design thickness of 53 µm. From this deposition several flat MLL segments have been manufactured. Some were coated with a stress layer inducing a well-defined bending deformation resulting in wedged MLLs [1]. Both types were then used for point focusing experiments at beamline ID 13 of ESRF. With focal spot sizes of 33 nm × 28 nm and 43 nm × 44 nm reconstructed by ptychography [2] both types have shown to produce nearly diffraction limited focusing for their respective apertures of 23 µm × 23 µm and 15 µm × 15µm.

The wedged MLL was also used for STXM measurements showing its great potential for user mode experiments at beamlines in the near future (see Figure 1). The total first order diffraction efficiency has been determined to be 19.8 % for the crossed wedged MLL system or to be 44.5% for each individual lens [3].

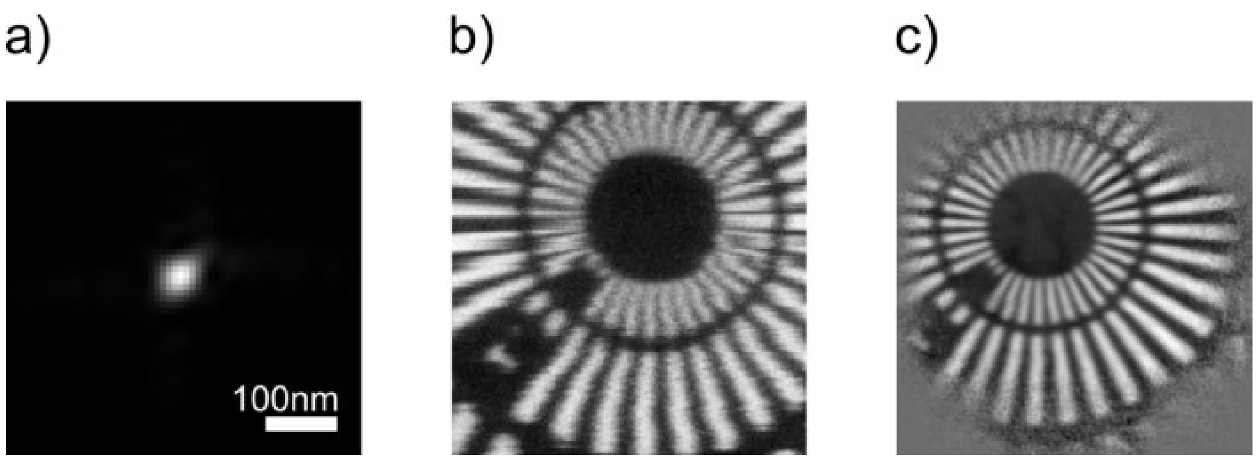

Figure 1: a) Reconstructed shape of the beam at the focus. b) STXM image of the center of a Siemens star with 50 nm lines and spaces. c) Reconstructed image of the same Siemens star by means of ptychography.

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Zone Plate Stacking for the Advanced Photon Source Upgrade Project

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The Advanced Photon Source Upgrade Project (APS-U) is planning to upgrade the APS storage ring to a multi-bend achromat magnet lattice that will produce an incredibly bright x-ray beam, advantageous for nanofocusing. Zone plates are diffractive nanofocusing optics similar to circular diffraction gratings but with changing period as a function of radius. The optical performance for hard x-ray zone plates is determined by the fabricated zones aspect ratio, zone height over zone width. Achieving the large aspect ratio (>20) for high quality hard x-ray zone plates is very difficult and sometimes impossible with current nanofabrication techniques. One strategy to increase the aspect ratio is to stack zone plates together, increasing the zone height of the total system, and near field stacking has been demonstrated at the Advanced Photon Source in the past [1,2]. More recently, a novel stacking regime has been proposed [3] and demonstrated [4] by stacking in the intermediate field. We will present our recent developments on stacked zone plate fabrication, intermediate field stacking apparatuses, and auto-alignment software development. The zone plate development program at the APS-U is tasked with developing nanofocusing systems suitable for future APS-U beamlines.

This research used resources of the Advanced Photon Source and Center for Nanoscale Materials, U.S. Department of Energy (DOE) Office of Science User Facilities operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

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Hard X-Ray Scanning Microscope Based on Refractive Optics

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The hard x-ray scanning microscope at beamline P06 of PETRA III at DESY in Hamburg serves a large user community, from physics, chemistry, and nanotechnology to the bio-medical, materials, environmental, and geosciences [1]. It has been in user operation since 2012, and is based on nanofocusing refractive x-ray lenses [2,3]. Using refractive optics, nearly Gaussian-limited nanobeams in the range from 50 to 100 nm can be generated in the range from 8 to 30 keV. The degree of coherence in the nanobeam can be made very high, allowing for ptychographic scanning coherent diffraction microscopy with highest spatial resolution [4]. With new types of refractive optics, such as adiabatically focusing lenses [5] and refractive lamellar lenses [6], the lateral size of the nanobeam can be reduced (e. g., to about 18 nm at 20 keV) and the efficiency improved. We demonstrate the performance of the coherent scanning microscope by imaging a polymer tandem solar cell in three dimensions [7]. After the current shutdown at PETRA III, the nanoprobe will be going into operation with an improved optics unit and differential interferometric position control of optics and sample. We will give an overview over the optical performance of the microscope and its applications.

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Recent Results and Future Plans for a 45 Actuator Adaptive X-ray Optics Experiment at the Advanced Light Source

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We report on the current status of the Adaptive X-ray Optics project run by Lawrence Livermore National Laboratory (LLNL). LLNL is collaborating with the Advanced Light Source (ALS) to demonstrate a near real-time adaptive X-ray optic. To this end, a custom-built 45 cm long deformable mirror [1,2] has been installed at ALS beamline 5.3.1 (end station 2) since September 2014. We will discuss general aspects of the instrument, present results from recent experimental campaigns and outline future plans for the project.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The document number is LLNL-ABS-667809.

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High-throughput Microtomography using Synchrotron Radiation at DESY

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The Helmholtz-Zentrum Geesthacht HZG is operating the user experiments for microtomography at the beamlines P05 and P07 at the storage ring PETRA III at the Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany. The beamline P05 is specialized on providing microtomography and nanotomography in the range of 5 to 50 keV [1]. The beamline P07 extends the energy range for microtomography up to 150 keV. During the first year of full user operation in 2013 attenuation and phase contrast techniques were successfully applied [2]. Within the shutdown of PETRA in 2014 main components for the microtomography system were changed. The software concept was redefined to fullfill the requirements for a standard data format which is defined and agreed within the Helmholtz Association in Germany and to allow for the realtime control of the experiment by including a GPU reconstruction pipeline [3]. A sample changing system was installed to increase the sample throughput. New detector systems were tested to provide data reduction and mathematics close to the detector [4].

Within this talk the actual status of the microtomography stations at P05 and P07 will be shown including the setup for grating based phase contrast. Several results obtained within user experiments will be given. The main focus will be on the software and hardware concept for high-throughput tomography. The new data format will become the base for the standardisation of microtomography applications using synchrotron radiation at DESY.

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Revealing Ultra-fast Processes in Real-time by Direct and Diffraction Hard X-ray Imaging

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The potential of hard X-ray imaging to tackle scientific questions especially related to materials sciences can be substantially increased when ultra-short time-scales are accessible [1]. Nowadays, unprecedented temporal resolution with hard X-ray imaging can be reached at synchrotron light sources thanks to high-speed CMOS cameras: with exposure times short enough to exploit the pulsed time-structure of the European Synchrotron Radiation Facility (ESRF), mechanically induced cracks in glass could be depicted [2]. By operating the storage ring of the ESRF in single-bunch mode with its correspondingly increased electron bunch charge density per singlet, the polychromatic photon flux density at insertion-device beamlines is sufficient to capture hard X-ray images exploiting the light from a single bunch (the corresponding bunch length is 140 ps FWHM). Hard X-ray imaging with absorption contrast as well as phase contrast in combination with large propagation distances is demonstrated.

Additionally, direct transmission with diffraction imaging has been combined successfully to track crack propagation in Si wafers in real-time: while an indirect detector acquired in radiographic mode images of the crack propagation by means of inline phase contrast an intensifier-based detector recorded in parallel diffracted images of the 220 reflex from the same event, giving information about the time evolution of the related strain [3]. The growth of strain fields in a hot Si wafer induced by water droplets is resolved from the moment before the cracks appear until they stop to propagate.

Future developments and their potential in the frame of the proposed Phase II of the ESRF Upgrade Program will be discussed.

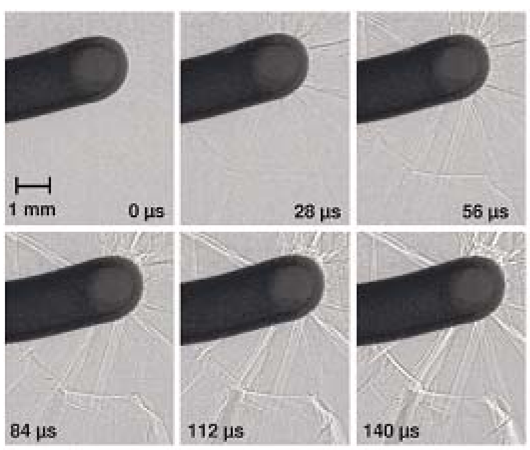

Figure 1: Time series recording crack propagation in a glass plate initiated by an accelerated bolt. The pictures are acquired via single-bunch imaging at beamline ID19 (ESRF).

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Large Area – high Speed Iron XANES Mapping of Impact Melt-bearing Breccias Utilizing a 384-pixel Maia Detector

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Metorite impacts are important geological processes which generate extreme pressures and temperatures over very limited spatial and temporal scales. These conditions result in the vaporization and melting of rocks, and, through this process impact melt rocks and breccias are created. Here, we report the use of a 384-pixel energy-dispersive Maia detector to obtain large-area, full spectrum iron XANES maps on impact melt-bearing breccias from the Mistastin Lake impact structure, northern Labrador, Canada. Figure 1a shows an iron µXRF map of a thin section taken from an impact melt-bearing breccia. The map was collected at the bending magnet based F3 beamline of CHESS using a Si (220) DCM focused with a single-bounce monocapillary optic. XANES mapping, from 7.05 to 7.3keV, was performed on an entire, 3.6 x 5.5 mm^2, area (see Figure 1) with 0.5eV energy steps in the near-edge region, an integration time of 15 milliseconds per pixel, and a spatial resolution of 40 µm, taking less than 8 hours to produce the entire XANES image stack with a flux of ~6×10^8ph/s. XANES spectra (Figure 1f) extracted from different spots in a glass clast and the surrounding breccia matrix show that iron has a homogenous phase distribution across the clast, and, that iron in the host matrix exhibits a different phase than iron within the clast. This suggests that iron in the clast is of different origin than iron in the main components of the breccia, which could provide insight into the dynamics of crater formation.

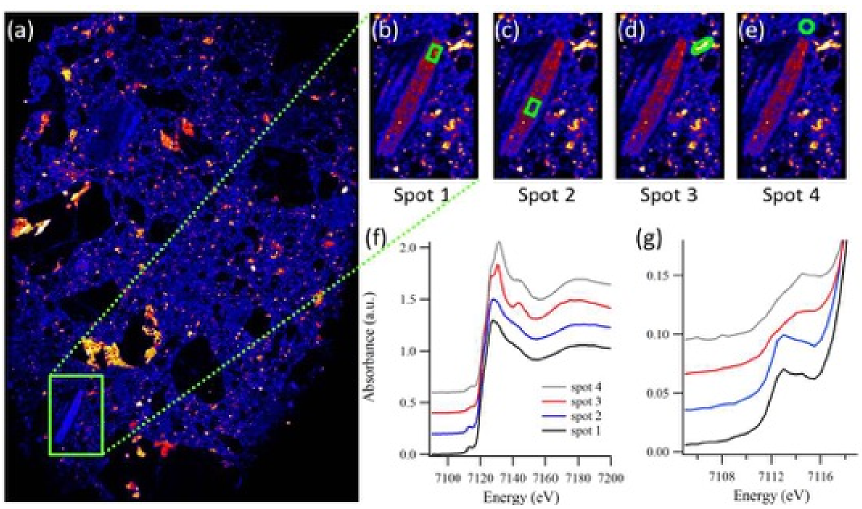

Figure 1 – Iron µXRF map (~25 x 36 mm^2) of an impact melt-bearing thin section (a), iron K-edge XANES mapping was performed inside the green box (3.6 x 5.5 mm^2); iron XRF maps from green boxed region in (a) showing spots (outlined in green) where XANES spectra were extracted from the image stack (b)-(e); iron K-edge XANES spectra of spots 1 – 4 (f), offset vertically for clarity; pre-edge region (1*s* → 3*d*) of iron XANES (g).

NanoXAS – Multichannel Imaging using Scanning Probe and X-Ray Microscopy

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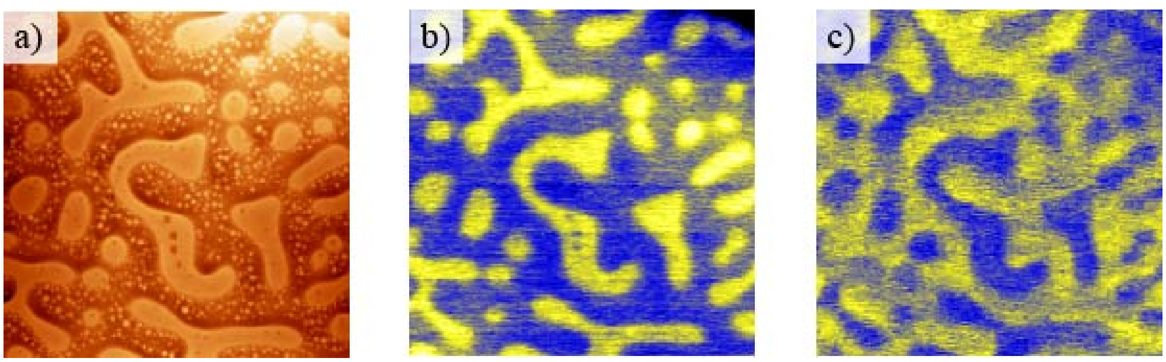
NanoXAS at the Paul Scherrer Institute is a soft X-ray beamline (270eV to 1800eV) dedicated to microscopy. The endstation is a completely novel setup allowing the user to perform scanning transmission x-ray microscopy (STXM) and scanning probe microscopy at the same sample location in a UHV environment and without complex alignment procedure [1]. In one hand STXM offers direct access to the local chemical composition, electronic structure, molecular orientation, order, and absolute density and, in the other hand, SPM can measure physical properties such as sample topography, elasticity, adhesion, friction, electrostatic and magnetic properties on lateral scales down to nanometers. To highlight the benefit of such multichannel approach some examples such as the characterization of organic films, the measurement of the density of very small amount of material [2] and the characterization of magnetic film will be presented.



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Scanning X-ray Microdiffraction Studies of Tissue Architecture

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Scanning x-ray micro-diffraction makes possible mapping of the distribution and variation of molecular architecture across a thin section of tissue at micron-scale resolution. A section of tissue 5-20 microns thick is scanned across a microbeam and a diffraction pattern is collected at each “pixel” on a grid with grid points 1-5 microns apart. Any feature of the diffraction pattern has the potential for acting as a 'contrast agent' that can be mapped across the grid of scattering volumes. For instance, characterization of a 250x250 micron field of view might utilize 2500 diffraction patterns taken with a 5 micron x-ray beam. Features in the x-ray patterns can be used to map the distribution of molecular constituents and their structural characteristics. The richness of the resulting data sets is significant and the potential applications extend to all complex materials. We have used the approach to characterize the variation in cellulose order, orientation and density in tissues within the stem of *Arabidopsis* and the distribution and structural form of amyloid deposits and neurofibrillary tangles in human brain tissue from subjects with Alzheimer's disease and Pick's disease. Here we present the fundamentals of the technique, the data processing packages developed to deal with the large volume of data generated and demonstrate the power of the approach in analysis of molecular architecture in these two examples.

Compton Scattering Imaging for Operando Observation of Lithium Batteries

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We propose an X-ray imaging technique based on synchrotron X-ray Compton scattering and demonstrate its application to batteries under in-situ and operando conditions.

Compton scattering imaging [1] is one of the in-situ techniques to observe the inside of large objects. This technique uses high-energy X-rays with high penetration power into an object and utilizes a feature that the intensity of Compton-scattered X-rays is proportional to the average electron density in the probing volume. The technique using synchrotron radiation was applied to a discharging coin cell [2], and it captured the migration of lithium ions in the positive electrode and revealed the structural change due to the volume expansion of the electrode. On the other hand Compton scattering is currently used to study electron momentum density distributions. The Doppler-broadened X-ray line due to the motion of electrons provides us information on the object [3]. By analyzing the line shape, it is possible to observe the lithium ion migration more precisely.

In this work we applied this technique to a PHEV (Plug-in Hybrid Electric Vehicle) battery and observed the change of a line-shape parameter due to lithium insertion and extraction in electrodes.

This work is supported by the Development of Systems and Technology for Advanced Measurement and Analysis program under Japan Science and Technology Agency.

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Serial Protein Microcrystallography Challenges

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Serial protein microcrystallography approaches developed originally at the LCLS XFEL source[1] are also revolutionizing protein microcrystallography at synchrotron radiation (SR) sources. While XFEL data collection is in principle radiation-damage free and performed at room temperature, the challenge for SR data collection is minimizing radiation damage and approaching room temperature data collection conditions. Raster-scanning diffraction by SR beams scaling in size with crystallite dimensions and fast-readout pixel detectors are a prerequisite.[2] I will discuss selected SR serial microcrystallography experiments to demonstrate progress along these lines showing that the same sample environments –such as LCP injectors-[3] and data reduction techniques[4] can be used for both sources. A common challenge of XFEL and SR serial crystallography is reducing sample consumption as far as possible. Indeed, manipulating and positioning of individual protein micro- and nanocrystals at predetermined positions allows a considerable reduction of sample consumption when using raster-scanning diffraction. I will discuss several approaches such as drop-on-demand systems and artificially structured surfaces which are of interest for scanning serial protein microcrystallography.

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NSLS-II Biomedical Beamlines for Micro-crystallography, FMX, and for Highly Automated Crystallography, AMX: New Opportunities for Advanced Data Collections

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We present the final design of the x-ray optics and experimental stations of two macromolecular crystallography (MX) beamlines at the National Synchrotron Light Source-II [1,2], to begin operation in early 2016. The microfocusing FMX will deliver a flux of ~5x10^{12} ph/s at 1 Å into a 1 – 50 µm spot, its flux density surpassing current MX beamlines by up to two orders of magnitude. It covers an energy range from 5 – 30 keV. The highly automated AMX is optimized for high throughput, with beam sizes from 4 – 100 µm, an energy range of 5 – 18 keV and a flux at 1 Å of ~10^{13} ph/s.

A focus in designing the beamlines lay on achieving high beam stability, for example by implementing a vertical axis double crystal monochromator at FMX. A combination of compound refractive lenses and bimorph mirror optics at FMX supports rapid beam size changes.

Central components of the in-house-developed experimental stations [3] are a horizontal axis goniometer with a 100 nm sphere of confusion, piezo-slits for dynamic beam size changes during diffraction experiments, a dedicated secondary goniometer for data collection from crystallization plates, and a next generation pixel array detector.

FMX and AMX will support a broad range of biomedical structure determination methods from serial crystallography on micron-sized crystals, to structure determination of complexes in large unit cells, to rapid sample screening and room temperature data collection of crystals in trays.

This work is supported by the US National Institutes of Health and the US Department of Energy.

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Serial Crystallography at PETRA III

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Structure determinations from microcrystals at low emittance synchrotron sources and X-ray free electron lasers (XFELs) [1] have recently attracted a great deal of attention and have the potential to change the way X-ray crystallographic data is collected from macromolecular crystals in the future. At new low emittance synchrotron sources such as PETRA III and NSLS II it has become possible to focus a large amount of photons into a spot of a few μm^2 only and hence to perform structure determinations from multiple microcrystals in a similar way as current serial crystallography experiments at XFELs.

At beamline P11 at PETRA III we follow two different approaches for sample delivery of the microcrystals to the X-ray beam. For room temperature measurements crystals are constantly delivered to the X-ray beam by flowing through a thin-walled capillary. The exposure time is determined by the flow speed in the capillary and the beam size. Another approach is the use of a micro-fabricated sample holder from single crystalline silicon with micropores (figure 1). Here a suspension of microcrystals is pipetted onto the chip and excess mother liquor is subsequently soaked off and crystals arrange themselves according to the micropore pattern. Data collection is performed by automatic raster-scanning of the chip at cryogenic temperatures. The two different approaches and their results will be presented.

In future we plan to extend these experiments to the time domain and to study irreversible biochemical reaction with micro-focus Laue crystallography using exposure times in the microsecond range.

Figure 1: CPV micro-crystals loaded onto the chip (left) and chip mounted on a standard crystallographic base (right).

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Upgrade of High Flux MX Beamline BL41XU at SPring-8

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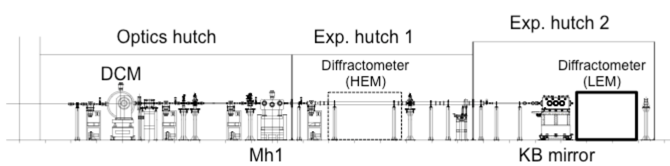
High flux macromolecular crystallography beamline BL41XU at SPring-8 started operation in 1997 and has contributed to structural determination of about one thousand of biological macromolecular structures [1]. However, the target for the structural study has been still getting more challenging. In order to cope with this situation, we have upgraded the focusing optics and the diffractometer last year.

The new optics adopts two-step focusing that consists of the first horizontal focusing mirror (Mh1) and KB-mirror. It achieved beam size of 2 μm × 2 μm - 35 μm (H) × 50 μm (V) with a photon flux of 1.7×10^{12} - 4.3×10^{13} photons/s at 12.4 keV. This is complementary specification with our microfocus beamline BL32XU [2]. One unique feature of this optics is to offset sample position and mirror angle to change beam size. The wide beam size range allows both micro-crystallography and high-resolution data collection that makes use of crystal volume. Newly installed detector PILATUS3 6M facilitated rapid data collection in combination with high flux beam. Together with the upgrade of the hardware, software tools that support diffraction based centering and helical data collection were implemented to make full use of the upgraded beamline. After the operation of one year, we have already obtained some fruitful results.

In addition to low-energy mode (LEM) thus described that covers 6.5 - 17.5 keV, we implemented high-energy mode (HEM) covering 20 keV to 35 keV. To allow HEM with minimum change of LEM setting, we have installed another diffractometer between Mh1 and KB-mirror.



Beamline layout after the upgrade

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A New and Novel Endstation for Microfocus Macromolecular Crystallography

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Beamline I24 at Diamond Light Source is a tuneable microfocus macromolecular crystallography beamline and has been in user operations since 2009. The current endstation has performed well for samples and beamsizes of ~10 µm and frame rates of order 10 Hz. On-going upgrades to the beamline will result in a reduction of the beamsize to ~2 µm × 4 µm, and this together with data collection at rates exceeding 100 Hz, necessitates the need for an upgrade to the endstation.

The new endstation at I24 comprises two goniometers at the sample position. A vertical high precision goniometer provides a sub-micron sphere of confusion for exploitation of microcrystals, while a horizontal goniometer allows data collection from crystals in crystallisation trays. The design allows automated exchange between cryo (vertical goniometer) and *in situ* (horizontal goniometer) modes, and automated sample exchange in both cases. Additional developments allow improved sample viewing, delivery of laser light to the sample position, and fast and accurate co-ordinated motions of multiple axes during the diffraction experiment.

An overview of the developments in instrumentation will be given, and experimental approaches that allow optimal collection of cryogenic and room temperature data collection from protein microcrystals described.

Micro-Focus Upgrade for the Macromolecular Crystallography Beamline X06SA at the Swiss Light Source

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The Macromolecular Crystallography beamline X06SA has been in operation at the Swiss Light Source since 2001 and is one of the most successful MX beamlines worldwide. Even though the diffractometer [1] and detector [2] have continuously improved over the years the optical layout, with a focal spot size of 85x10um^2, has remained unchanged.

The original one-stage focusing optics at the beamline X06SA, was very limited in shaping the phase space (beam size and divergence) since they can only image the X-ray source once. To take advantage of the existing excellent adaptive X-ray optics [3, 4], a two stage-focusing scheme was developed. In this design, the existing (primary) optics focuses the source to an intermediate position, which serves as a secondary source, which is then re-focused with a pair of Kirkpatrick-Baez mirrors [5] located very close to the sample. In order to gain maximum control of the phase space, the secondary source point is designed to be movable in a two-meter range and dynamic focusing is required for both primary and secondary focusing elements. The design goal is to reach variable beam size from 2 x 1 um^2 to 100 x 100 um^2 and beam divergence from 0.5 mrad to parallel. To fully exploit these beam properties in macromolecular diffraction experiment, the next generation pixel-array detector EIGER 16M [6], featuring smaller pixel, higher resolution, faster frame rate and negligible readout time, will be installed in summer 2015. The optics design and commissioning, the first results from EIGER 16M detector and the first users' experiments will be presented.

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Direct Observation of Bond Formation by Femtosecond X-ray Solution Scattering

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Thu

Plenary

8:30 AM

The time-resolved X-ray solution scattering measurement at storage ring has achieved successfully in investigating the structural dynamics of excited states including solvents and solutes, however its time-resolution has been limited to about 100 picoseconds. To understand the whole photochemical reaction, it is essential to observe not only relatively long-lived pico- and nanosecond excited states which play an optical functional role including the strong emission from triplet exciplex and the photocatalytic activity but also the growth process of the functionality on more early timescale. Therefore, chasing the molecular deformation in femtosecond time scale is crucial to unravel the reaction mechanism and rate of the transition state. In this study, the time-resolved X-ray solution scattering using femtosecond X-ray pulses [1] is conducted to give the entire picture of photoreaction of a luminescent gold oligomer. Ultrafast molecular deformation, which had been suggested by ultrafast absorption spectroscopy and theoretical calculation, is directly observed with atomic resolution. The formation of covalent bond is clearly observed as the molecular deformation immediately after photoexcitation, and the generation of larger oligomer is obvious evidence of the adjustability of exciplex emission [2].

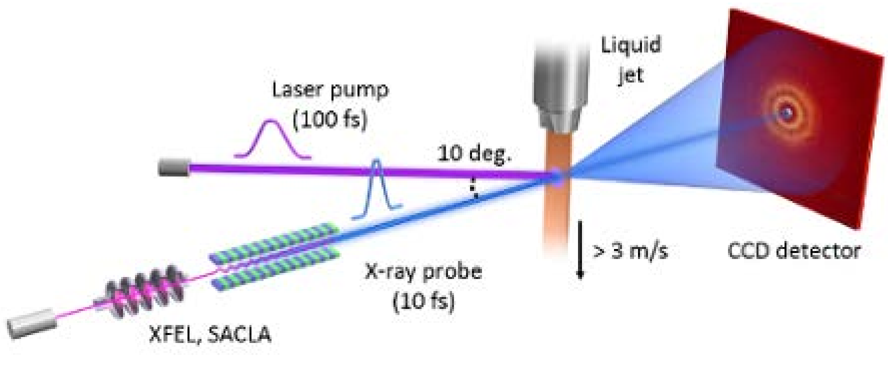

Fig. 1 The experimental setup of the femtosecond X-ray solution scattering at the XFEL facility, SACLA.

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Seeded Free-Electron Lasers and Applications

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While early Free-Electron Lasers (FELs) were "seeded" by external laser fields, the lack of short wavelength sources resulted in the development of X-ray FELs operating from the Self-Amplified Spontaneous Emission (SASE) process, which starts solely from shot noise. However, the limited longitudinal coherence of SASE FELs limits both FEL performance and user experiments, prompting efforts to push the seeding process down to the X-ray regime. In this talk I will describe the current state of short wavelength seeded FELs around the world, future directions in seeded FEL research, and highlight a few successful user experiments with seeded FELs.

Development of Ptychographic Tomography for Scientific Applications

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There are many examples of natural and artificial materials where a hierarchical organization of their 3D structure spanning many length scales leads to special properties. Typically different techniques need to be combined for the visualization of such materials. For large sample volumes in the millimetre range micro computed tomography provides an overview of the inner structure, while subsequent electron microscopy can ultimately reveal the detailed structure with high resolution. However, the latter requires sectioning, which can produce artefacts and may destroy features one aims to characterize. Imaging non-destructively a piece of material of up to 100 micron thickness with high resolution would enable detailed visualization of its 3D structure on the mesoscale, providing in some cases a direct input for simulations of the material properties. However, providing this capability remains a challenging task.

X-ray ptychography is a high-resolution imaging technique in which the specimen is scanned in transmission using a confined, coherent illumination, in such a way that the illumination overlaps in subsequent scanning steps [1]. At each position, the scattered coherent diffraction intensity is recorded in the far field and iterative phase retrieval algorithms are employed to reconstruct the complex-valued transmissivity of the specimen, yielding a direct image with both absorption and phase contrast. Similarly to other coherent diffraction imaging methods, the resolution in ptychography is limited by the largest angle at which intensities can be reliably recorded, and it can be much better than that limited by the illumination size or by the scanning step size. Moreover, ptychographic algorithms are very robust to reconstruct projections of thick objects with phase variations exceeding many times 2π, while preserving a very high sensitivity. Therefore, the combination of many such phase projections at different incident angles of the beam via tomographic reconstruction provides high quality and quantitative 3D density maps with resolutions down to a few tens of nanometers [2-4].

The implementation of ptychographic tomography as a tool for science is challenging. In practice ptychographic reconstructions rely on the illumination remaining unchanged during the entire scan, resolution is ultimately limited by the accuracy of the positioning of the specimen with respect to the illumination, and considerable efforts must be undertaken to perform the scanning at high speed. Furthermore, ptychographic reconstructions need to match the speed of the acquisition, and this also applies to the processing of images for tomographic reconstruction. Here we present how the development of acquisition strategies, algorithms and instrumentation has enabled the use of ptychographic tomography as a reliable tool to address scientifically relevant questions [5,6]. As an application case we present a recent study to investigate the density of calcium silicate hydrates in hydrated cement paste [7,8].

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The Multi-Bend Achromat Storage Rings

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Not very long time ago, the successful 3rd generation storage ring technology was judged to be mature. Most of the 3rd generation sources used the Double-Bend (DBA) or Triple-Bend (TBA) concept. It was however a well-known fact that increasing the number of unit cells in the storage rings was a powerful way of increasing the ring brilliance, but at the prize of increasing the size and cost of the rings. On top of that, the associated reduction of the dynamic aperture of the rings was of great concern.

The Multi-bend Achromat concept, including a miniaturisation of the ring elements has now drastically changed this picture. The MBA rings, now under construction or planned, offers orders of magnitudes higher brilliance compared to conventional technology. Several light sources around the world are now planning to implement or actually implementing this Multi-Bend Achromat (MBA) concept.

This presentation touches on the science drivers for increasing the source brightness. We will then discuss the MBA concept as well as its advantages and challenges. A short survey of the MBA activity around the world will also be presented.

As an example, the MAX IV project will be described; its concept, history, current status, schedule and beam-line plans.

The FERMI Seeded-FEL Facility: Status and Perspectives

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The FERMI Free Electron Laser (FEL) in Trieste, Italy [1] operates in the extreme ultraviolet and soft x-rays (EUV-SXR) wavelength range delivering high-fluence, stable, ultra-short pulses [2,3]. Its unique design based on a seeded scheme and on tunable undulators allows unprecedented control of pulse parameters such as wavelength, phase, polarization, synchronization, pulse duration and implementation of multi-color FEL schemes [4–7]. Both FEL-1 and FEL-2 lines with nominal wavelength range 100–20 nm and 20–4 nm, respectively, are open to users. We report on the unique features of FERMI, in particular those that have evolved beyond the original design, and on their application to pioneering experiments [7,8]. We also present the upgrades that are planned to further expand the capabilities of this unique light source.

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NSLS-II Storage Ring Insertion Device and Front-End Commissioning

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The National Synchrotron Light Source II (NSLS-II) is a state of the art 3 GeV third generation light source at Brookhaven National Laboratory. In the spring 2014, the storage ring was commissioning up to 50 mA without insertion device. In the fall, the project beamlines, includes seven insertion devices on six ID ports were commissioned within two and a half months. These beamlines consist of IXS, HXN, CSX-1, CSX-2, CHX, SRX, and XPD-1, from the radiation sources elliptically polarizing undulator (EPU), damping wiggler (DW) and in vacuum undulator (IVU) to cover the VUV through the very hard x-ray range. In this paper, a number of commissioning and operation experiences are discussed here, such as injection, lifetime, ID residual field and compensation, source point stability, beam alignment and tools for control, monitor and beam protection.

Status of the ESRF's New Low-Emittance Storage Ring

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The ESRF has now started all the work to implement, in 2019, a completely new Storage Ring that will substitute the present one. This new Ring will have its horizontal emittance reduced by a factor 30. This will benefit the Light Source Users with increases of the brilliance, up to a factor 100, and the transverse coherence fraction for the X-rays.

We will review the main constraints and requirements for the new ring, to satisfy all existing beamlines by keeping their present source-points and by continuing to serve all filling modes at 200mA nominal current. The main characteristics of the Hybrid-Multi-Bend lattice are described that resulted from a process of many iterations aiming at their optimization while also improving the feasibility for the magnet and vacuum systems.

The magnet system, with a total of 1088 elements, features now octupoles, high gradient quadrupoles and sextupoles, and 7 dipole magnets per cell. The latter include combined dipole-quadrupoles and longitudinal-gradient-dipoles, with some of these being obtained with permanent magnet structures. For some magnets prototype testing has now started.

The design of all vacuum chambers is progressing and prototypes of bellow structures have been validated. Ray-tracing and power distribution studies are progressing and soon allowing the precise design of all absorbers. Detailed simulations of the vacuum conductance have been concluded.

The orthogonal heptapod design is retained for the girders, and a prototype is soon delivered. The strategy for the challenging work and requirements of pre-assembly, pre-alignment and the transport and installation is in progress.

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New Capabilities at CHESS

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The penetrating power of x-rays in the 30 – 150 keV range is critically important for perfroming *in situ* and *operando* studies of functional, structural, and engineering materials and devices. Yet, worldwide, there are only five synchrotron light sources - those powered by high-energy (E>5GeV) storage rings – that provide intense undulator x-ray beams in this energy range. High-energy storage rings are expensive to build; therefore, continuous up-grades to the storage ring, the insertion devices, the x-ray optics, and the end-station capabilities are essential. Over the past year, CHESS has reconfigured its storage ring, installed novel compact permanent magnet undulators, installed novel (broadband) x-ray optics, and developed sophisticated end-station capabilities (detectors, collimators, environmental chambers) to support specific research programs. By matching source characteristics, focusing, bandwidth, collimation and detector properties for optimal system performance and working closely with leading research groups to address specific research challenges, CHESS has created experimental capabilities that deliver unique scientific value to the worldwide community of synchrotron users. In this talk, I will review several of the novel technologies developed at CHESS and the forefront science that they enable.

PETRA III: Experiments at a Low Emittance 6 GeV Synchrotron Radiation Source

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Since 2010 the storage ring PETRA III at the research center Deutsches Elektronen-Synchrotron (DESY) is operated with 6 GeV particles as a low-emittance 3rd generation synchrotron radiation source, today exhibiting the smallest emittance of all high energy X-ray sources [1]. Up to now 300m of the storage ring, which corresponds to roughly 1/8 of the circumference, have been equipped with undulator beamlines adding up to 35 experiments out of which 15 can be run simultaneously. At PETRA III DESY targets for an average of 4000 h/a X-ray beam for the user program. At present more than 2000 individual scientists visit the facility each year. In 2015 and the following years, more experiments (up to 12 independent stations) will be available.

The extraordinary small emittance stimulated the installation of unique experiments with record values of beam foci, coherence volume and flux. Consequently, DESY launched new science groups with topics closely related to synchrotron radiation, which in collaboration with the beamline scientists and engineers work on scientific projects pushing the X-ray methods to new limits. Additionally, PETRA III has attracted many external institutions and universities which provided large grants for instrumentation and staff.

This contribution highlights the extraordinary parameters of PETRA III and gives an overview of the experiments, the user activities and examples of in-house sciences. A short outlook to the PETRA III extension project is also presented.

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Future of ePix Detectors for High Repetition Rate FELs

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Free electron lasers (FELs) made the imaging of atoms and molecules in motion possible, opening new science opportunities with high intensity x-ray laser pulses. The first hard x-ray FEL (LCLS at SLAC) entered operation in 2009[1]. Some new or upgraded FEL facilities will operate at high repetition rates in the order of tens to hundreds of kHz (*e.g.*, LCLS-II, European XFEL). The high repetition rates require corresponding upgrades of current FEL detectors (and other instrumentation).

Different detectors are considered for requirements of particular experiment types and beam structures. Some of these detectors are being designed or developed by the SLAC detector group to match the high repetition rate FEL requirements. Most detectors under consideration can be used for single pulse imaging or accumulate over many frames.

For imaging, fast ePix100[2,3] and ePix10k[4,5] detectors are under development, allowing linear detection of up to 800 keV and 80,000 keV respectively per pixel per frame. These detectors can be equipped with high-Z sensors for high energies (over 18 keV). For spectroscopic detection, ePixS detectors are being considered.

The different high repetition rate experiment types and their detector requirements will be discussed, together with the corresponding detector performance and readout optimization.

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Recent Developments of the THz Streak Camera at PSI for FEL Temporal Diagnostics

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SwissFEL is the upcoming free electron laser facility at PSI, aiming to provide users with X-ray pulses of lengths down to 2 femtoseconds. A new device based on terahertz streak camera concept is currently built at PSI for measuring the relative arrival time and the length of FEL pulses [1]. By using clustered xenon pulses in the chamber high resolution measurements can be performed with hard X-Ray photons.

This conference contribution presents the theory behind the measurement technique [2, 3] and some simulations of the streaking process. It also discusses the first measurements obtained with a prototype setup, the pulse arrival time and length monitor (PALM), carried out at SACLA with photon energies up to 12.4 keV [4].

The results from the first measurements point the way towards improvements, which will be incorporated in the new design. We will utilize a twin interaction region to measure streaked and un-streaked photoelectron spectra. This will allow us to compensate for the shot-to-shot jitter of the spectral width, improving the resolution of the photon pulse length measurement. The contribution discusses the effect of the Gouy phase shift on the resolution and proposing a way of increasing the dynamic range of the detector for measuring the pulses with large arrival time jitter.

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High Channel Count X-ray Spectroscopy Detector for X-ray FELs

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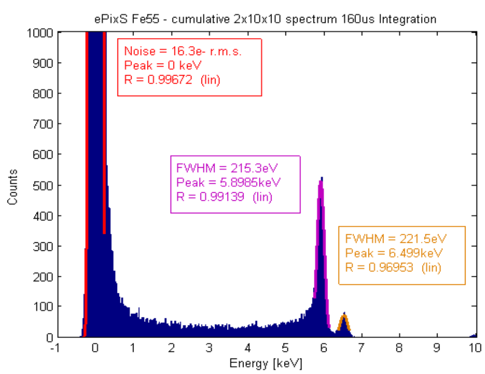
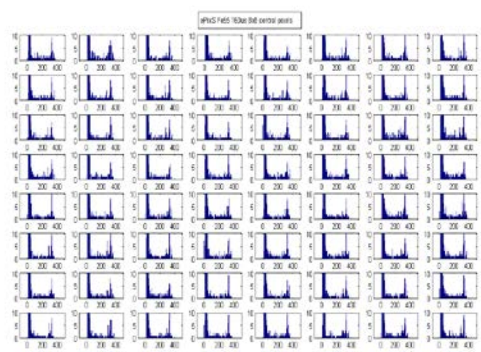
SLAC has developed a novel detector platform that can deliver Silicon Drift Detector (SDD) like performance at strobed x-ray sources, where it can measure a spectrum on a pulse-by-pulse basis. It is based on a hybrid architecture with p-i-n diodes flip-chipped bonded to mixed signal integrated circuits.

The integrated circuit ePixS is part of a SLAC family of x-ray signal processors named ePix [1,2] and has an equivalent input noise of 8 e^- r.m.s. at room temperature. The full detector is typically operated at -40° C where it shows a noise floor of 12 e^- r.m.s. at nominal 32µs integration time. The first generation offers 4 100x100 pixel sub-arrays with a pitch of 500 µm. The system is scalable to 10,000 pixels. Since it has an electronic shutter, sub arrays can be phased to achieve a 100 kHz effective frame rate.

Although intended for LCLS, it is a natural fit to the requirements of other FELs and offers significant advantages over an array of SDDs in terms of cost, data acquisition, and system complexity, while providing similar spectral resolution. The large channel count drastically reduces the time required to take a two or three-dimensional scan of a sample allowing more experiments to be performed in a given period. The detector can be also operated at synchrotrons, showing performances comparable to SDDs in terms of resolution and, because of the high segmentation, with higher throughput. The system and results from prototypes will be presented.



a)



b)

Fig. 1a: Fe^{55} radioactive source spectrum over 2 entire chips at -40° C. Noise from sensor leakage is the main component, thus increasing with longer integration times from 8 e^- r.m.s. at 32µs to 16 e^- r.m.s. at 160µs. The current design is optimized for synchronous applications; meaning that with a pulsed FEL the peak-to-background ratio improves dramatically. A new asynchronous version of ePixS is under design optimized for applications outside FELs. Fig. 1b: Spectra on multiple channels.

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AreaDetector Framework for SwissFEL On-line Burst Mode Diagnostics

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The SwissFEL is a new generation X-ray free electron laser (XFEL) under construction at the Paul Scherrer Institute (PSI) in Villigen, Switzerland [1]. This light source represents one of currently four XFEL projects which deliver hard X-ray pulses at wavelengths down to 1 Å. We discuss the software control for XFEL diagnostics setups for which the EPICS-toolkit is providing flexible control system architecture. The setup is rigorously based on the *SynApps* "Positioner-Trigger-Detector" anatomy [2,3]. On the “Detector” side an increased effort was made to standardize the control within the EPICS *areaDetector* (AD) software framework [4]. In this contribution we focus on image snapshots taken by array detectors which need to be tagged by bunch markers for later cross-correlation with other XFEL parameters. A specialized AD-plugin was developed to stream data between data-acquisition server and data-analysis clients hosting popular Python-based AD-plugins [5]. The HDF5-compliant and lossless high-performance transfer of detector data between server and client is based on the ØMQ embeddable networking library [6]. The ØMQ data-stream includes metadata with bunch-markers, used for on-line analysis inside dedicated client AD-plugins. We demonstrate that in typical setups the system operates lossless within the data acquisition rate of 100 Hz imposed by the SwissFEL design. Within the server-client system, a burst-mode capture of image data with consecutive on-line analysis is processed with unambiguously tagged shot-to-shot markers. The concept opens the doors towards high performance scientific data services in SwissFEL experiments, such as realizing on-line shot-to-shot wavefront [7] or spectrometry [8] diagnostics.

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The Soft X-ray Monochromator Beamline at the European XFEL: Design and Expected Performance

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A challenge of transporting coherent femtosecond pulses produced by novel XFEL (X-Ray Free-Electron Laser) sources of unprecedented brilliance is addressed in design of the soft X-ray beamline at the European XFEL facility. The beamline shall transport pulses produced by the SASE3 undulators in a range 0.25 – 3 keV; these pulses are grouped in trains of up to 2700 pulses delivered at up to 4.5 MHz repetition rate within train containing up to 10^{14} photons in pulse. To tackle damage problem and minimize deterioration of spatial and temporal beam properties, the beamline has been designed as hundreds meters long, the length of reflective optical elements comprising the beamline reaches 850 mm, and quality of optical surfaces set to 50 nrad slope error and 2 nm PV residual height error. A plane variable-line-spaced grating monochromator will serve to provide monochromitized beam. Expected to serve multipurpose user community, the monochromator would allow controlling temporal pulse properties and will be operational in both low and high resolution modes. The beamline performance has been evaluated from the point of view of energy resolution, transmission, as well as spatial and temporal properties of transported FEL pulses. Wavefront propagation methods, indispensable in case of coherent beams, have been used in addition to analytical estimates.

Soft X-Ray Ptychography of Nano-Materials at the Advanced Light Source

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The Advanced Light Source has developed an ultra-high resolution x-ray microscope based on soft x-ray ptychography for the study of nano-materials and has demonstrated the world's highest spatial resolution [1,2]. The microscope is capable of three dimensional as well as spectroscopic imaging at around 10 nm spatial resolution. It utilizes an ultra-stable scanning mechanism with laser interferometer feedback for sample positioning, a fast frame rate charge-coupled device detector [3] for soft x-ray diffraction measurements and can achieve scan rates of up to 100 Hz, including motor move, data readout and x-ray exposure, with a positioning accuracy of better than 2 nm RMS. A low numerical aperture zone plate lens provides a well-defined x-ray probe on the sample with a long depth of field which is well suited for applications such as tomography and *in situ* measurements which require a long working distance. We have implemented a high performance data pipeline that enables real time ptychographic reconstructions on a distributed computational architecture with a user friendly interface [3,4]. This instrument, called The Nanosurveyor, can achieve a spatial resolution nearly 20 times finer than the x-ray spot size in both two and three dimensions using x-rays with energies up to 2 keV. Using this instrument on a bending magnet beamline, we demonstrate chemical composition mapping of $LiFePO4$ nano-particles in two and three dimensions at a spatial resolution unmatched by conventional microscopes. Once moved to the new Coherent Scattering and Microscopy beamline it will enable spectromicroscopy and tomography of materials with wavelength limited spatial resolution.

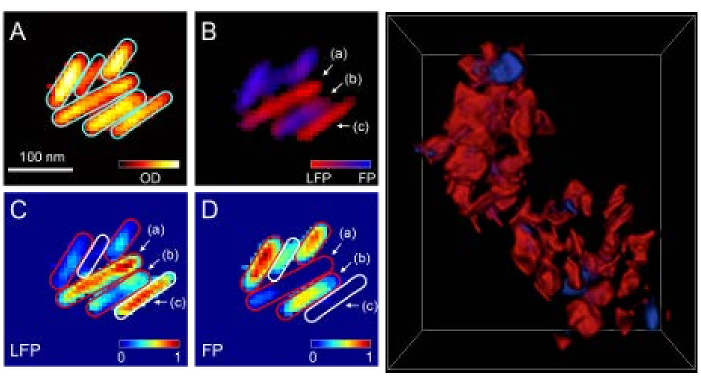

Figure 1. Spectro-ptychography of partially delithiated LiFePO4 using the Nanosurveyor. (A) Optical density of LiFePO4 crystals with ~20 nm width. (B) Chemical composition map showing FePO4 (blue) and LiFePO4 (red). (C-D) Heat maps showing content of the end members of the phase transformation. (E) Three dimensional chemical map of a similar sample. The pixel size and voxel size are 6 nm.

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Micro- and Nanoimaging at the Diamond Beamline I13L – Imaging and Coherence

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The Diamond Beamline I13L is dedicated to imaging on the micron- and nano-lengthsale, operating in the energy range between 6 and 30 keV [1]. For this purpose two independent stations have been built.

The imaging branch is fully operational for micro-tomography and in-line phase contrast imaging with micrometre resolution. Currently a full-field microscope providing 50nm spatial resolution over a field of view of 100µm is tested. On the coherence branch coherent diffraction imaging techniques such as ptychography and coherent X-ray diffraction are currently developed.

The beamline contains a number of unique features. The machine layout has been modified to the so-called mini-beta scheme, providing significantly increased flux from the two canted undulators. New instrumental designs have been employed such as a robot arm for the detector in diffraction experiments. The imaging branch is operated in collaboration with Manchester University, called therefore the Diamond-Manchester Branchline.

Beamline instrumentation and the related science will be presented, the latter covering a large area including bio-medicine, materials science, chemistry geology etc.

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Ptychography Operated in Fly-scan Mode

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Ptychography is a scanning version of coherent diffractive imaging method [1]. It is rapidly merging as a robust analytical tool for characterizing the transmission function of the sample and the wavefront of the illumination beam. Due to its intrinsic scanning nature, the throughput of ptychography is limited by the data collection efficiency. With a regular step-scan scheme, moving and settling motors accumulate up to nearly 50% of the total experiment time. We report a method to eliminate this motion overhead by adapting fly-scan concept into ptychography data collection and analysis [2]. We demonstrate that the data collection efficiency can be raised to over 80%, while the reconstructed image quality is well preserved. This approach significantly increases throughput of this scanning imaging technique. It is a critical development for applications on three-dimensional visualization and imaging dynamic systems.

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X-ray Scattering from Optically Trapped Nanoparticles

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Coherent X-ray diffraction imaging (CXDI) is a sensitive microscopic method for imaging crystalline and non-crystalline objects at high spatial resolution. CXDI is particularly useful for measuring strain patterns in nanoparticles that arise due to the large ratio of surface area to bulk volume. A major barrier to applying CXDI to freestanding micro- and nano- scale objects is their tendency to freely move within the intense beam from synchrotron source. Typically such objects must be securely bonded to a substrate, which can alter their internal structures. While the forces moving such particles are not completely understood, we believe optical tweezers may be a solution.

Optical tweezers provide a unique method to control small particles, ranging from several microns to tens of nanometers. Using optical techniques, these laser trapped particles can be manipulated, and forces on the objects in the trap can be measured. Combined with phase modulation techniques, optical traps with different geometries and polarization can be generated, resulting in an accurate orientation of anisotropic particles [1,2].

We are attempting to measure the x-ray diffraction from optically trapped micro- and nano- particles of both materials and biological sample origin with the goal of eliminating the barrier to studies of free standing objects due to uncontrolled sample drifts. We have observed the Bragg peaks from optically trapped ZnO nanowires by utilizing a homebuilt dynamic holographic optical tweezers which are compatible with CXDI at beamline-34 of APS. We are improving the stability of trapped particles and optimizing conditions and techniques for recording coherent diffraction images. During our next beamtime we will use CXDI to image a free standing sample for the first time.

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Speckle Based X-ray Phase Contrast and Dark-field Contrast Imaging

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X-ray phase contrast imaging can substantially enhance the sensitivity for samples with weak absorption over the conventional absorption contrast imaging, whilst dark-field imaging techniques can provide supplemental and inaccessible information with sub-pixel resolution. A number of X-ray imaging techniques have been proposed and developed in the last two decades. However, most of these imaging techniques are typically limited due to the requirement of either sophisticated experimental conditions or stringent beam properties. We present a novel approach which is based on X-ray speckles coupled with simple experimental arrangement; and it can produce both phase and dark-field contrast images simultaneously by using a sheet of inexpensive abrasive paper [1-3]. The speckle based technique can be classified into single image mode and speckle scanning mode. The single image mode is highly desirable for rapid in-vivo imaging or computed tomography (CT) with moderate spatial resolution. We have demonstrated that the speckle based phase contrast CT can yield quantitative volumetric information on the real part of the refractive index by acquiring only a single image per projection[2]. In contrast, the high resolution phase and dark-field signal can be extracted by scanning the speckles along single directions. We have recently demonstrated that the speckle based directional dark-field imaging show great potential for the study of strongly ordered systems[1]. Moreover, unprecedented angular sensitivity in the nanoradian range has been achieved for this speckle based technique by decreasing the scan step size and / or increasing the detector distance[4]. We foresee a widespread use of this technique in the near future, and we expect it to open the way for X-ray multi-mode imaging for both biomedical applications and material science.

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Highly Automated Solution SAXS at EMBL Hamburg

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Small Angle X-ray Scattering (SAXS) on biological macromolecular solutions gained considerable interest over the last decade. SAXS became an important part of the "toolbox" of numerous structural biologists [1] and there is a well-established and rapidly growing user community utilizing the SAXS instrument dedicated to biological solution scattering technique.

The EMBL BioSAXS beamline P12 (third generation PETRA-III ring, Hamburg) was built to succeed the popular X33 BioSAXS beamline [2] (second generation DORIS-III ring) and tailored for biological solution SAXS [3]. The low instrumental background allows for data collection on weakly scattering samples. Sensitive and often scarce biological samples are reliably and rapidly handled by a sample changer. Size exclusion chromatography columns and additional spectrometers (UV/Vis, RALS, and refractometer) can easily be connected to the beamline for on-line purification and sample characterization.

Particular care was taken to automate the measurements such that they can be performed with minimal input from the user. Fully automatic data collection is made possible by a sample changer robot. The SASFlow pipeline analyzes the data directly after collection and provides the users with the overall parameters of the solutes and low resolution shapes within minutes on data take. The high level of automation permitted to perform over 85,000 measurements during the last full beamyear and also allows for remote and mail-in measurements.

The characteristics of the instruments and main sample environment will be described and additional experiments exploiting the high flux (time-resolved SAXS) and small size (microfluidics) of the beam will also be discussed.

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CrystalDirect: A New Crystallization Plate and Automated Crystal Harvester to Benefit from the Power of Future X-ray Sources

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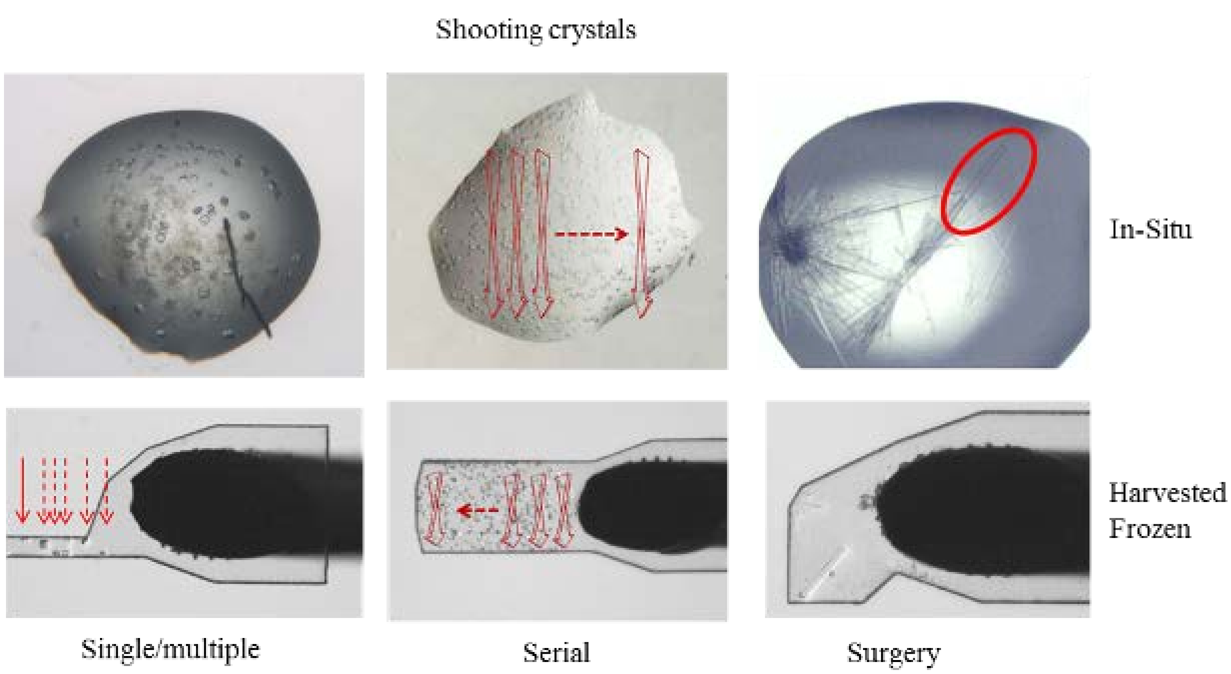
Over the last decade automation has dramatically increased the efficiency of the processes involved in macromolecular x-ray crystallography. Nevertheless, the connection between crystallization and x-ray data collection suffers from the need of transferring crystals from the support where they grow to supports used to optimally collect x-ray diffraction data. Furthermore, some projects require applying chemical treatments to the crystals before data is collected. To facilitate the automation of these delicate and time consuming operations we have developed CrystalDirect (CD) (1), a system based on a new crystallization plate where crystals grow on an ultra-thin film directly compatible with x-ray data collection and are recovered by laser photo ablation. CD allows harvesting crystal of any size without mechanical stress, separately or in batches. Optional treatments can be applied directly in the plate, before harvesting.
CD enables the integration of crystallization and data collection and opens new opportunities for crystal treatment, in-situ data collection, micro-crystallography and serial data collection. The CD plates and harvester will be presented as well as new goniometer setups for in-situ data collection. The potential of CD when linked to a crystallization LIMS will be shown and a plan for integrating a harvester at beamline drawn.



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Towards Automatic Data Collection Pipeline for Membrane Protein Structure Analyses at Beamline BL32XU

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Recently, crystal structure analyses of membrane proteins are dramatically accelerated as crystallization methods are improved. Among the methods, so-called lipidic cubic phase crystallization, referred to as LCP method, is of great use for obtaining crystals with higher diffracting power. However, crystals from the method are usually tiny and surrounded by thick lipids. Thus, a micro-focus beamline is suitable for data collection with good signal-to-noise ratio using LCP crystals.

The beamline BL32XU at SPring-8 is dedicated to the protein micro crystallography. The beamline can provide 1~10 µm beam with the flux density of 10^{10} photons/sec/µm^2, and high impact structure analyses of membrane proteins had been achieved with the micro beam[1-5]. All of these structures were solved with SAD/MAD from a single or a few crystals with so-called helical data collection method.

Though the helical data collection still works well for larger crystals than a few tens of µm, multiple crystal strategy, obtaining dataset from multiple tiny crystals, is essential for more efficient structure analyses at higher resolution. We are now developing a pipeline of data collection from multiple tiny protein crystals. The system includes crystal mounting, automatic loop centering, diffraction raster scan, crystal positioning, data collection strategy, data reduction and merging. We will demonstrate a performance of the system by showing several experimental results of membrane structure analyses.

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Native SAD Phasing for Routine Structure Determination

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Native SAD uses the weak anomalous scattering of light elements naturally present in the target macromolecule (P, S, Cl, K and Ca). Its success relies on very accurate measurement of the reflection intensities, which is limited by both random and systematic errors, as well as radiation damage, and crystal quality. The merging of data sets from multiple crystals [1] can yield high quality anomalous data but requires statistically equivalent crystals whose availability may be limited. A new strategy [2], which consists in collecting multiple low-dose data sets on only one crystal entity in multiple orientations, was successful on 20 real-life cases, including a 266 kDa multiprotein complex, the largest structure solved by native-SAD to date. Developed at beamline X06DA-PXIII at the Swiss Light Source, the method benefits from very stable X-ray source and optics, a high-precision multi-axis PRIGo goniometer [3] and a readout noise-free PILATUS detector calibrated for low energies.

Here, we will present the key features of our anomalous data collection strategy, which effectively reduces instrumentation errors. First results on SmarGon (from SmarAct GmbH), the commercial multi-axis goniometer based on PRIGo, as well as on the new hybrid photon counting detector EIGER (from Dectris, Ltd.) will be presented as well.

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The Macromolecular Crystallography Beamline BioMAX at the MAX IV Laboratory

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The MAX IV storage ring facility [1] at the MAX IV Laboratory, Lund, Sweden, will come into operation in mid 2016. The MAX IV 3 GeV ring [2,3] will be the first storage ring using a multibend achromat lattice with sub-nm rad emittance [4]. The BioMAX beamline [5] is one of the first phase beamlines and is dedicated to macromolecular crystallography. Thanks to the exceptionally low emittance of the storage ring, 0.3 nmrad, the focused beam will both be small, 20 x 5 μm^2, and have a low divergence, 0.1 x 0.1 $mrad^2$. With the 18 mm period in-vacuum undulator with 4 mm minimal gap (Neomax Materials, Japan) the flux at the sample will be $>10^{13}$ photons/s. The optics of the beamline are relatively simple with a liquid nitrogen cooled Si(111) horizontally deflecting double crystal monochromator and a Kirkpatrick-Baez mirror pair (FMB Oxford, UK). Both mirrors have one-moment benders (Winlight X, France). This design allows work with both small crystals and large biomolecular complexes. BioMAX is designed to be flexible and serve a broad range of needs for the structural biology community.

The experimental environment will be equipped with an MD3 diffractometer (Arinax, France) with a vertical main rotation axis and two additional axes in a mini-kappa geometry. The setup will include state-of-the-art area detector, sample changer, beam conditioning equipment and auxiliary equipment such as sample cryo and fluorescence detector.

The beamline will be controlled using MXCuBE [6] version 3 developed in collaboration with six other facilities in Europe. MXCuBE will be integrated in the MAX IV standard environment with Tango and Sardana. The beamline will offer remote access, will use ISPyB [7] for sample management and will have a highly parallel analysis environment for on-line analysis of data.

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Taiwan Photon Source: Current Status and Future Perspectives

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Recently the National Synchrotron Radiation Research Center (NSRRC) in Taiwan has successfully constructed a low-emittance 3 GeV synchrotron light source, the Taiwan Photon Source (TPS). The TPS with a circumference of 518 m is composed of 24 double-bend achromatic (DBA) cells connected by six 12-m straight sections and eighteen 7-m straight sections. The natural emittance of the TPS is 1.6 nm · rad with a small dispersion in the straight sections. Figure 1 displays an aerial photograph of the NSRRC. The TPS commissioning proceeded at a speedy pace, delivering its first synchrotron light in December 2014 and the electron current in the storage ring reaching 100 mA in March 2015. In phase-I operation, the TPS will use two sets of KEK-B type superconducting RF cavities to achieve an electron current of 500 mA in a top-up injection mode.

Seven TPS phase-I beamlines are currently under construction. Taking full advantage of the high-brilliance TPS, these beamlines aim for the forefront of research, covering the diverse photon sciences in an energy range from soft to hard X-rays. These beamlines are optimized for protein micro-crystallography, low-energy excitations of novel materials with atomic specificity, spectroscopy and diffraction on the submicron and nanometer scales, scattering of coherent X-rays, and scanning nanoprobe studies that will resolve structures with a 40-nm spatial resolution. Six of the seven phase-I beamlines are scheduled to be completed by the end of 2015 and to be available for users in mid 2016.

In this talk, we will first share our experiences of the TPS construction and commissioning with the synchrotron community. The design and the construction of the seven TPS phase-I beamlines will also be presented, followed by an overview of future beamline plan.

Figure 1: An aerial photograph of the NSRRC. This photograph shows the recently completed TPS and the 21-year-old Taiwan Light Source (TLS). The proposal of the TPS project was prepared in 2004. The civil construction of the TPS began in February 2010, and was completed in December 2013. The assembly of main accelerator parts was completed in 2014. The TPS project came to realization after ten years of efforts.

Status of the European XFEL

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The European X-Ray Free-Electron Laser (European XFEL) is currently under construction in the Hamburg metropole area, Germany. First electrons have been generated in the laser-driven injector and commissioning of the injector with beam will commence in fall 2015. The production and installation of the main accelerator, provided to a large extent through contributions by partners from a large number of countries, is in full swing. The 91 undulator segments to be installed for the various FEL sources have been produced, tested and await installation. The challenging x-ray optics and diagnostics elements are under procurement and first components have been installed in the tunnels. The design of the science instruments is largely complete and installation has started. This important activity will continue until start of operation. Further important developments concern the x-ray detectors, synchronized optical lasers, sample environments and the data acquisition and storage systems.

By summer 2016 the accelerator construction and installation will be completed and commissioning with beam will be started at an electron energy of 17.5 GeV. By the end of 2016 the electron beam and the undulators shall be ready for first generation of FEL radiation. In 2017 the commissioning of the electron beam, the undulator and FEL operation, and of the science instruments will continue. The three FEL sources and the six science instruments will be taken into operation in the sequence SASE1 – SASE3 – SASE2 over a period of about four to six months. In parallel, in 2017 first user experiments will be performed. Full performance of accelerator, FEL radiation and science instruments shall be reached in 2018. It is currently planned to increase the hours for accelerator operation dedicated to the user program from 1000 hrs in 2017, over 2000 hrs in 2018, to the final 4000 hrs in 2019.

In the presentation the current layout of the facility and of the science instruments will be discussed. Major instrumentation efforts will be presented and an outlook to the commissioning of the facility and the initial science program is provided.

Status of SwissFEL, the X-ray Free-electron Laser at PSI

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SwissFEL is a hard x-ray FEL facility that is currently under construction at PSI. It is based on an S-band injector that generates a 320 MeV electron beam at a 100 Hz repetition rate. In a linear accelerator based on C-band technology the electron energy will be increased up to 5.8 GeV. In-vacuum undulators with a period of 15 mm are then used to produce a photon beam in the wavelength range between 0.1-0.7 nm. In a next stage, the facility will be extended by a second undulator line that will cover the soft x-ray range between 0.7-7 nm, and by using Apple III undulators, full polarization control will be possible. A fast switch-yard will allow a simultaneous operation of both FEL-lines at a 100 Hz repetition rate.

The construction activities started in spring 2013 and in spring 2015, first components were already installed inside the new building. In summer 2015, the installation of the injector will begin, and it is planned to commission the injector in spring 2016. Later in 2016, it is foreseen to commission the main linear accelerator and the hard x-ray undulator line. The installation of the soft x-ray FEL is planned to start in 2018.

This presentation will give an overview of the project status and the related R&D activities.

Thu

Parallel

3:30 PM | *New Facilities II* | Thu-E

Current Status of PLS-II Beamlines

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Pohang Light Source(PLS) in Korea has been upgraded to PLS-II with the storage ring energy of 3 GeV, electron beam emittance of < 10 nm rad, 400 mA top-up mode, and 20 insertion beamline ports [1]. The upgrade was carefully planned with an International Advisory Committee. PLS had one year shutdown in 2011 and the ring current has been slowly increased from 100 mA(year 2012) to 300 mA(year 2014) in top-up mode. To fully exploit those upgraded properties, beamlines have been constructed for ultra-small angle scattering, wide energy range x-ray absorption fine structure(XAFS), nano XAFS, soft x-ray nanoscopy, coherent x-ray scattering, electron-beam-synchronized time-resolved scattering, etc.; some are being constructed for μ-macromolecular crystallography, spin-resolved angle-resolved photoemission spectroscopy, etc. The beamline specifications, science results and/or future plans of those beamlines are to be presented.

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FLASH2: Operation, Beamlines, and Photon Diagnostics

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FLASH2 is the major extension of the soft X-ray free-electron laser FLASH at DESY. A new undulator line is situated in a separate accelerator tunnel and driven additionally by the FLASH linear accelerator. This turns FLASH into a multi-beamline FEL user facility. FLASH2 is equipped with variable-gap undulators and can deliver a wavelength largely independent of FLASH1 to user endstations. First lasing of FLASH2 was achieved in August 2014 with simultaneous user operation at FLASH1.

The new FLASH2 experimental hall offers space for up to six user endstations, some of which will be installed permanently. Two beamlines will cover the short wavelength range with a fundamental in the water window and a 5th harmonic down to 0.8 nm, while up to four beamlines will cover the longer wavelengths of 6 - 40 nm and beyond. This wide wavelength range, while of high interest to users, is challenging from the instrumentation point of view. Photon diagnostics have been developed for many years at FLASH and are in routine operation. Online, often pulse-resolved measurements of intensity, position, wavelength, wavefront, and pulse length as well as photon beam manipulation tools such as a gas absorber and filter sets have been optimized for FLASH2. Pump-probe facilities including a state of the art optical laser, a dedicated undulator beamline for THz radiation and split-and-delay units offering XUV-XUV pump-probe capabilities complete the FLASH2 user facility.

Sirius: The New Brazilian Synchrotron Light Source

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The Brazilian Synchrotron Light Laboratory (LNLS) is building Sirius, a 4th generation 3 GeV storage ring based on a five bend achromat (5BA) magnetic lattice that will be able to reach 270 pm rad emittance (bare machine). The low beta straight sections of this 518 m circumference storage ring have been optimised for maximum photon brilliance (betax,y ~ 1.5 m) and to allow new configurations of insertion devices (with small BSC < 5 mm (v) x 8 mm (h)). Phase 1 and 2 will include 13 beamlines, 8 based on insertion devices, and 5 based on the high-brilliance 2T superbends at the center of each 5BA .The ID beamlines will cover experimental programs, such as coherent diffraction imaging of large cells, tender x-ray nano-spectroscopy, matter under extreme conditions, soft-x-ray spectroscopy of solid/liquid/gas interfaces and high energy tomography, while more conventional synchrotron techniques will make use of the 2T superbends. In this talk I will overview some of the main aspects of this new storage ring and its first beamlines, as well as the current status of the project. Please use Times new Roman 11 pt., line distance 1.0 and a margin of 2.5 cm or 1inch on the inside margin on each side of the page and on the outside margin on each side of the page (as is set in this template), justified text. Please leave one blank line before each sub-heading and a distance of 6 pt after each sub-heading and text paragraph. The final decision on key-note, oral or poster presentations will be made by the program committee.

X-ray Optics for the European XFEL

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X-ray lasers produce very collimated and coherent X-ray pulses. Within one pulse of several femtoseconds, the number of photons produced is often comparable to photons per second available at synchrotron experiments.

For X-ray lasers driven by a super-conducting linear accelerator high repetition rates of X-ray pulses can be reached -- in the case of the European XFEL up to 4.5 MHz during a 600 microsecond long pulse train.

X-ray optics faces the challenge to transport these beams that produce extreme and cycling heat loads without destroying the optical elements or deteriorating the beam quality. At the European XFEL optical elements are typically placed 300 meters and more from the source point, which reduces the power density, but increases the sensitivity to vibrations. Solutions and ongoing developments tailored to these requirements on mirrors, monochromators and safety devices will be presented.

New Reflection Zone Plate Array Optics with Individual Depth Profiles for Ultra-fast X-ray Applications

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Variably polarized ultra-short X-ray radiation pulses in the soft X-ray spectral range are optimal tools for exploring ultrafast phenomena in condensed matter. Such experiments conducted at storage ring or HHGs require high transmissive optics. Therefore, new optical concepts based on reflection zone plate arrays (RZPA) that preserve the photon flux, pulse length and polarization [1] have been installed and successfully used in user operation at *Femtoslicing* facility at BESSY II and HHG facility [2].

Here, we report about the design and performance of a new generation of RZPAs for experiments with 100-fs time resolution at the upgraded *Femtoslicing* facility. Aiming at minimum losses in x-ray flux at reasonable resolution, we proposed and used an RZPA as a single optical element for diffraction and focusing in the zone plate monochromator which has been successfully used in user operation since 2012. Now, we designed and fabricated the next generation of RZPAs: three-dimensional RZPAs with individual profile depths for each specific lens to further gain the flux by increased diffraction efficiency up to 20%. Eleven Fresnel lenses, designed for the energies from 410 to 1900 eV were fabricated on the same substrate.

This work was financially supported by Marie Curie FP7-Reintegration-Grants within the 7th European Community Framework Program (PCIG10-GA-2011-297905), the BMBF project “Next generation instrumentation for ultrafast X-ray science at accelerator-driven photon sources” (05K12CB4) and the FemtoSpeX project (05K10PG2).

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Demonstration of Feasibility of X-ray Pump–x-ray Probe Experiments using a Hard X-ray Split-and-delay Optics Combined with Focusing Mirrors

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A hard x-ray split-and-delay optics (SDO), which provides two x-ray pulse replicas with time delay precisely controlled, based on the Bragg diffraction using Si(220) crystals was developed (Fig. 1) for x-ray pump–x-ray probe (XPXP) experiments at x-ray free-electron laser (XFEL) facilities, such as SPring-8 Angstrom Compact free-electron LAser (SACLA) [1]. This SDO consists of two thin (≈10 μm) crystals acting as beam splitter/merger [2], two thick crystals (beam reflectors), and two channel-cut crystals (total six), and covers a wide range of photon energies 6.5–11.5 keV. Time delay between the replicas depends only on difference in path length between the upper and lower branches (jitter-free) and is controlled by translating the beam reflector crystals linearly along each 2θ axis. The replicas are recombined collinearly, that enables combination with a Kirkpatric-Baez (K-B) mirror system. This is of great advantage for XPXP experiments because combination with such a kind of focusing system can realize high power density fields up to 10^{20} W/cm^2 [3]. In that case, spatial overlap of the replicas on focal (sample) plane is necessary in order to collect effective datasets in XPXP experiments.

We demonstrated the feasibility of XPXP experiments in combination with a K-B mirror system at BL29XUL of SPring-8 in Japan [4]. After adjustments of the crystal components, 2D focal profile with slight displacement (<30 nm) was obtained. Efficiency measured was ≈27% (13% upper and 14% lower), much higher than reported one (≈0.3% [5]). We present the details of this experiment and results of characterization at SACLA.

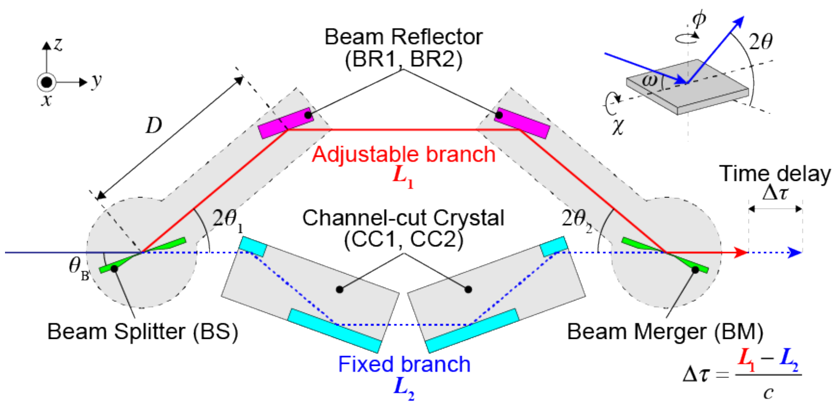

Fig. 1
Schematic configuration of a hard x-ray split-and-delay optics based on the Bragg diffraction using Si(220) crystals.

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Development and Throughput Simulations of a Hard X-ray Split and Delay Line for the MID station at the European XFEL

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The Materials Imaging and Dynamics (MID) instrument at European XFEL[1,2] aims at the investigation of nanosized structure and nanoscale dynamics using coherent radiation. Applications to a wide range of materials from hard to soft condensed matter and biological structures are envisaged. Here, we present an X-ray split and delay line (SDL) for MID and Fig. 1 sketches its design. The SDL provides pairs of output X-ray pulses with a variable time delay ranging from -10 fs to 800 ps in an energy range from 5 to 10 keV. The output beams can be either collinear or have an offset angle between them. In the latter case (inclined mode), the two beams can overlap at the sample by using an additional mirror and the offset angle results in spatially separated diffraction patterns on the detector. This SDL will allow studying fast dynamics using, for example time-resolved X-ray Photon Correlation Spectroscopy (XPCS)[3], X-ray Speckle Visibility Spectroscopy (XSVS)[4], ultrafast X-ray tomography[5], and temporally and spatially resolved X-ray holography[6]. Moreover, not only X-ray pump X-ray probe experiments, but also X-ray probe - optical pump - X-ray probe (XOX) and optical pump - X-ray probe - X-ray probe (OXX) experiments will be enabled through a proper synchronization with an external optical laser. To address the throughput of the SDL we have performed simulations with SASE pulses and Fig. 2 presents the results. 1.1% and 3.5% throughputs in mono and two-color modes are obtained, respectively. Simulations of the SDL performance with self-seeded pulses are in progress.

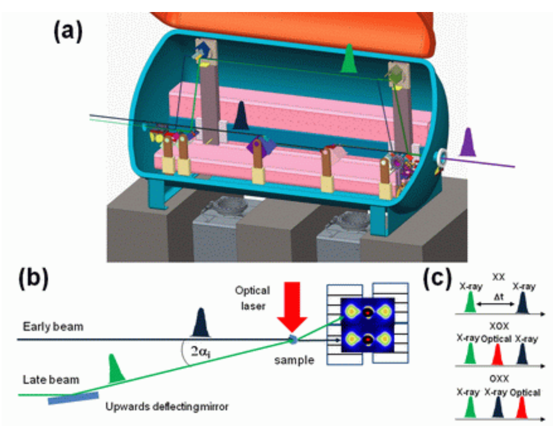

Fig. 1 (a) Mechanical design of the SDL. The time delay is obtained by splitting the beam and sending one part on trajectory which is longer (upper branch) than the distance travelled by the other half of the beam (lower branch). The SDL operates with Si(220) perfect crystal reflections as the main optical element. (b) Sketch of the inclined operation of the SDL and (c) the possible pump-probe schemes.

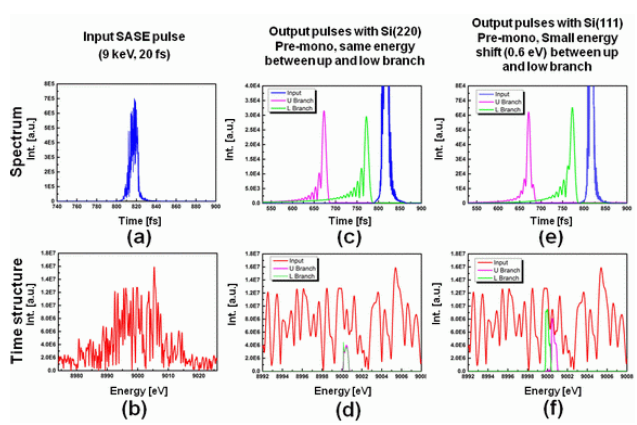

Fig. 2 Throughput simulations of the intensity splitting scheme with SASE pulses. (a) and (b) are the energy spectrum and time structure of the input pulse, respectively. (c) and (d) are output pulses with a Si(220) pre-mono, same energy at upper (U) and low (L) branches. The total throughput is ~1.1%. (e) and (f) are output pulses with a Si(111) pre-mono and a small energy shift (0.6 eV) between upper and lower branches. In this case the total throughput is ~3.5%.

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Diamond Single Crystal Optics for Seeding at High Repetition Rate X-ray Free Electron Lasers

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The lasing process in SASE X-ray Free Electron Lasers produces extremely powerful, ultra-short and fully transversely coherent X-ray pulses. However, the process starts from electron-gun noise, which imposes a broad, spiky spectrum and poor temporal coherence of emitted XFEL radiation. The latter shortfalls can be overcome by self-seeding scheme using bandwidth filtered X-rays from upstream undulator segments to provide a seed for downstream segments. At the European XFEL facility, hard X-ray self-seeding will be produced by a diamond single crystal monochromator. Existing monochromator setups cannot accommodate severe heat load due to high repetition rate of European XFEL. We therefore plan to employ a new design based on direct contact between two polished single crystal diamond plates, with a 0.1 mm thick high quality crystal plate used as the actual monochromator, and the other acting as a holder and heat sink. Here we report on an experimental study of the new setup carried out at ESRF beamline ID06, with white undulator radiation used to provide heat loads comparable with average power of XFEL beam pulse train. The gradient of diamond crystal deformation was measured as a function of heat load to determine the threshold values for tolerable heat load, transport and deformation range parameters. The results show that implemented design is deformation-free, mechanically stable and capable of accommodating power levels exceeding the peak value expected for European XFEL self-seeding. In the presentation, we shall discuss the impact of irradiative and conductive heat transfer on cooling efficiency and further setup improvements.

Optical Design of the Aramis-beamlines at SwissFEL

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SwissFEL is a free electron laser facility for hard and soft X-rays located at the Paul Scherrer Institute research center in Switzerland. The construction of the first hard X-ray "Aramis" branch started in 2013. After its completion in 2016 it will deliver photons in the wavelength range from 1 Å to 7 Å into two beamlines. The Aramis 1 beamline is dedicated to ultrafast photochemistry and serial femtosecond crystallography, whereas the Aramis 2 beamline will specialize in pump probe crystallography on solid-state samples. A third beamline, Aramis 3, is planned for phase 2 and is dedicated to material science and nano-crystallography.

The contribution briefly introduces the SwissFEL facility and continues with a detailed view on the beamlines. Various operational modes and the expected performance are presented.

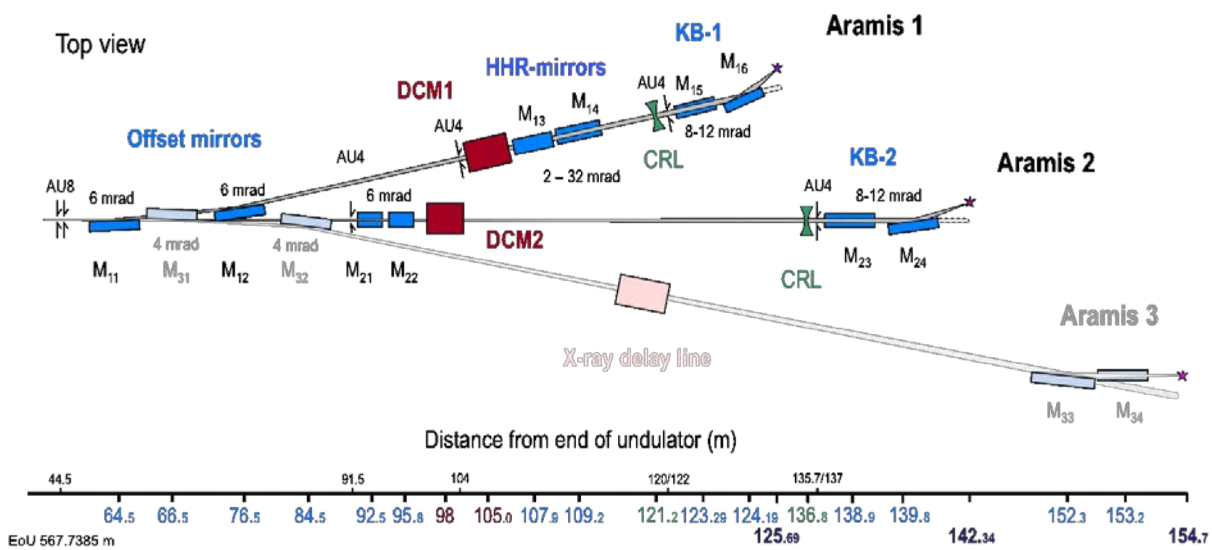

Fig. 1: Layout of the hard X-ray beamlines at Aramis.

Chemical Speciation Imaging at Environmentally Relevant Concentrations using X-ray Fluorescence Microscopy

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X-ray fluorescence microscopy (XFM) [1] can be used for elemental and chemical microanalysis across length scales ranging from millimeter to nanometer. XFM is ideally suited to quantitatively map trace elements within whole plant and other biological specimens, environmental and soil samples. The elemental sensitivity of the X-ray fluorescence probe provides valuable information for investigations in a diversity of environmental sciences, and the high penetration of hard X-rays enables measurement of whole cells and tissue sections with a minimum of preparation.

Advances in X-ray fluorescence detection methods such as the Maia detector [2, 3] now enable high definition images at mega-pixel per hour rates. The ability to rapidly acquire 2D images enables 3D information such as fluorescence tomography to be obtained in realistic times. Chemical speciation (valence) imaging (CSI) is a 3D technique where the third dimension is spectroscopic detail [4]. CSI produces an X-ray Absorption Near Edge Structure (XANES) spectra from the X-ray fluorescence signal at each pixel in the spatial image. Fitting of spectra with incident X-ray beam energy tracking has been developed in GeoPIXE software using the Dynamic Analysis method [5, 6].

CSI has been demonstrated at the Australian Synchrotron XFM beamline [1] with micron resolution and moderate definition (10K pixels) across a diverse range of sciences and applications from environmental chemistry [7] to arsenic toxicity in crop production [8]. Recent studies probing and optimising the efficiency and sensitivity of the CSI technique to achieve measurements at environmentally relevant concentrations will be presented.

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A Hard X-ray Nanoprobe Beamline and Electron Microscopy Facility at Diamond Light Source

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Beamline I14 is the hard X-ray nanoprobe beamline currently under construction at Diamond Light Source, UK, and scheduled to come into operation in 2017. The beamline will be a dedicated facility for nanoscale microscopy and micro-nano SAXS, serving two endstations housed in a new external building approximately 175m from the main synchrotron ring. The nanoprobe endstation aims to achieve the smallest possible focus (initial aim 50nm) with the capability to exploit future optics developments. The optical design will be optimised for scanning X-ray fluorescence, X-ray spectroscopy and diffraction. The mesoprobe endstation will be optimised to carry out small and wide angle X-ray scattering studies as well as scanning fluorescence mapping with a variable focus beam in the range 5μm – 100 nm. The beamline will complement electron and optical microscopy and enable new science in a number of areas spanning materials science, biology, engineering and earth science.

The I14 beamline facility has also been merged with a new national electron microscopy facility providing 4 electron microscopy suites covering the physical and life sciences. This facility combines staff and expertise from a number of different areas which we believe we will allow us to make exciting progress in sample preparation techniques and correlative x-ray and electron microscopy studies. Here we present the design and key specifications of Beamline I14, and highlight potential applications.

Operando Soft X-ray Scanning Photoelectron Emission Microscopy for Graphene FETs and Organic FETs

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In order to investigate pin-point electronic structure in nano-devices during device operation, we have developed soft X-ray scanning photoelectron emission microscopy (SPEM) with an angle-resolved electron analyzer (VG Scienta R3000) where different voltages can be independently applied to five electrodes for gate, source, drain and ground in a sample holder connected to a semiconductor parameter analyzer. The spatial resolution was determined to be 70 nm [1]. The first example is graphene FET with a back gate structure for nano-spectroscopy. The line profile analysis of the C 1*s* peak (sp^2) in the channel across the graphene-Ni electrode interface clearly demonstrates the existence of the charge transfer region [2]. Furthermore, we discovered the linear band dispersion in graphene FET for the first time by direct observation of *p*-type doping feature under back gate biasing [3]. The second example is organic FET. The *operando* nano-spectroscopy revealed the real time change in potential profiles in the channel of organic FET during gate and drain biasing [4], where *p*-type doping and potential profiles in the channel from source to drain are in good agreement with the drain current change upon gate biasing, and potential profiles calculated by gradual channel approximation (GCA), respectively. Thus, *operando* nano-spectroscopy can provide us with useful information for improving performances of nano-devices.

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Versatile Tool for nm-scale Spatial Resolution X-ray Imaging using MLL Nanofocusing Optics

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The Hard X-ray Nanoprobe (HXN) beamline at NSLS-II has been designed and constructed to address scientific and technological questions at the nm-scale. The HXN X-ray Microscope is a key instrument for the beamline, providing a suite of experimental capabilities which include scanning fluorescence, diffraction, differential phase contrast and ptychography techniques, utilizing Multilayer Laue Lenses (MLL) and zoneplate (ZP) as nanofocusing optics. To provide broader impact and explore the phase space in materials research studies, the instrument is equipped with a temperature regulation system capable of varying specimen temperature between 100 K and 1000 K. During this presentation, different phases of the x-ray microscope development process will be discussed, various prototype systems designed and constructed will be reviewed [1-2]. Preliminary data demonstrating nm-scale spatial resolution imaging using MLL optics will be presented [3].

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X-ray Nanodiffraction Meets Materials Science

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The Nanofocus Endstation of beamline P03 (PETRA III, Hamburg) is operated jointly by Helmholtz-Zentrum Geesthacht and the University of Kiel, and is one of the very few synchrotron endstations providing the experimental conditions for scanning X-ray nanodiffraction. This technique, in turn, is an excellent tool for materials science. It readily serves structural information with sub-micrometer spatial resolution from crystalline and semi-crystalline materials (metals, biomaterials, synthetic compounds) for the retrieval of e.g. grain orientation, residual stress profiles, crystal structure or texture. Because of the long focal distance focusing, the wide energy range of the P03 beamline and the highly adaptive sample positioning system, high resolution nanodiffraction experiments can be performed even in extended sample environments. A beam size of typically 350 nm * 250 nm is used for these experiments and is generated using a long focal distance KB-mirror focusing system.

The strong focus on materials science at P03 is demonstrated by the wide range of experiments already performed with in situ sample environments: pressure, indentation force, tensile stress, fluid shear, magnetic fields - all of these parameters were successfully modified in situ and combined with the high spatial resolution provided by nanofocused beam [1-6].

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Taking Advantage of a Confocal Microprobe Setup Specifically for Optimizing Micro-beam X-ray Absorption Spectroscopy

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X-ray absorption spectroscopy can often prove problematic when performed in a microprobe or nano-probe environment. When samples are relatively thick, there can be questions regarding whether elements, or specific chemical forms of elements, and structural features within the sample are truly co-localized. When samples are thin sections, then signal to noise suffers when the total integrated signal from a detector can become dominated by sources of scatter extraneous to the sample. Addition of a focusing optic to the detector to create a confocal detection volume overcomes these difficulties for both thicknesses of samples. A greater degree of confidence with respect to co-localization can be expressed and the scatter contribution to the total integrated detector signal is now limited to scatter only produced from within the confocal volume. Examples shall be presented making use of polycapillary and newer micro-channel focus array [1] detector optics, and a case shall be presented for routine use of a confocal microprobe setup for microbeam spectroscopy even when the x-ray fluorescence images are taken in a standard non-confocal mode.

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Multiplexed High-resolution Imaging Spectrometer for Resonant Inelastic Soft X-ray Scattering Spectroscopy (RIXS)

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Resonant inelastic soft X-ray scattering spectroscopy (RIXS) has been demonstrated as one of the most powerful techniques for studying the elementary excitations and electronic correlations in complex materials. However, small photon cross-section subjects this technique to the trade-off between throughput and energy resolution. The energy compensation concept developed for AGM-AGS RIXS setup at NSRRC demonstrated that the off-axis radiation from a synchrotron beamline can effectively be used to enhance the detection throughput. However this setup inherently integrates the excitation photon energies. Therefore it is not suitable for RIXS measurements that focus on the excitation energy dependence.

The optical design of a two-dimensional imaging soft X-ray spectrometer is developed [1]. The beamline monochromator will produce a dispersed spectrum in a narrow vertically illuminated stripe (~2 microns wide by ~2 mm tall) on the sample. The spectrometer will use Wolter mirrors to image the extended field on the sample in the incident photon energy direction (vertical). At the same time it will image and disperse the scattered photons in the orthogonal (horizontal) direction. The ability to image the flat-field illumination of the sample onto the flat field of the detector enables recording the RIXS map – displaying the RIXS features with respect to excitation (vertical) and emitted (horizontal) energies. Our design shows that this spectrometer can provide a completely parallel resonant inelastic X-ray scattering measurement at high spectral resolution (~ 30000) over the energy bandwidth (~ 5eV) of a soft X-ray absorption resonance. The scientific applications of this multiplexing RIXS will be discussed.

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High-contrast Sub-millivolt Inelastic X-ray Scattering for Nano- and Mesoscale Science

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Photon and neutron inelastic scattering spectrometers are microscopes for imaging condensed matter dynamics on very small length and time scales. Inelastic x-ray scattering permitted the first quantitative studies of picosecond nanoscale dynamics in disordered systems almost 20 years ago. However, the nature of the liquid-glass transition still remains one of the great unsolved problems in condensed matter physics. It calls for studies at hitherto inaccessible time and length scales, and therefore for substantial improvements in the spectral and momentum resolution of the inelastic x-ray scattering spectrometers along with a major enhancement in spectral contrast.

Here, we report the first implementation of a conceptually new inelastic x-ray scattering spectrometer, based on new principles of x-ray monochromatization and spectral analysis [1]. We prove the efficacy of combining novel optical components to create an ultra-high-resolution inelastic X-ray scattering (UHRIX) spectrometer with unmatched performance in terms of energy, momentum resolution, and spectral contrast. The UHRIX spectrometer features a spectral resolution function with steep, almost Gaussian tails, sub-meV (0.62 meV) bandwidth and improved momentum resolution. UHRIX opens up uncharted space on the dynamics landscape. This is precisely the space of vital importance for the science of disordered systems. UHRIX is perfectly optimized for latest generation of the synchrotron radiation sources and x-ray free electron laser facilities. We have successfully verified the new spectrometer concept by carrying out measurements on liquid glycerol in previously inaccessible regions of energy and momentum transfer, and achieved very promising results.

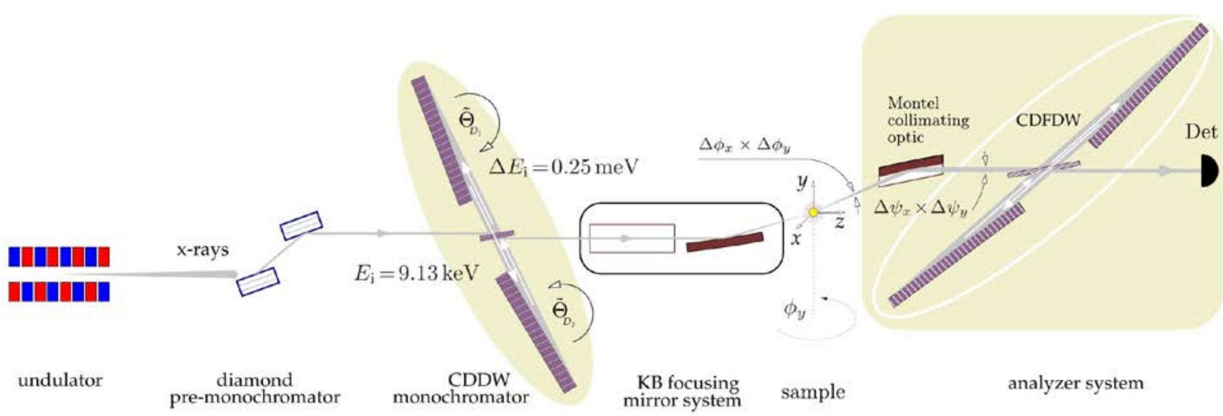

Figure: Layout of the UHRIX instrument.

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Dual-Array Valence Emission Spectrometer (DAVES): A New Approach for Hard X-ray Emission Spectroscopies

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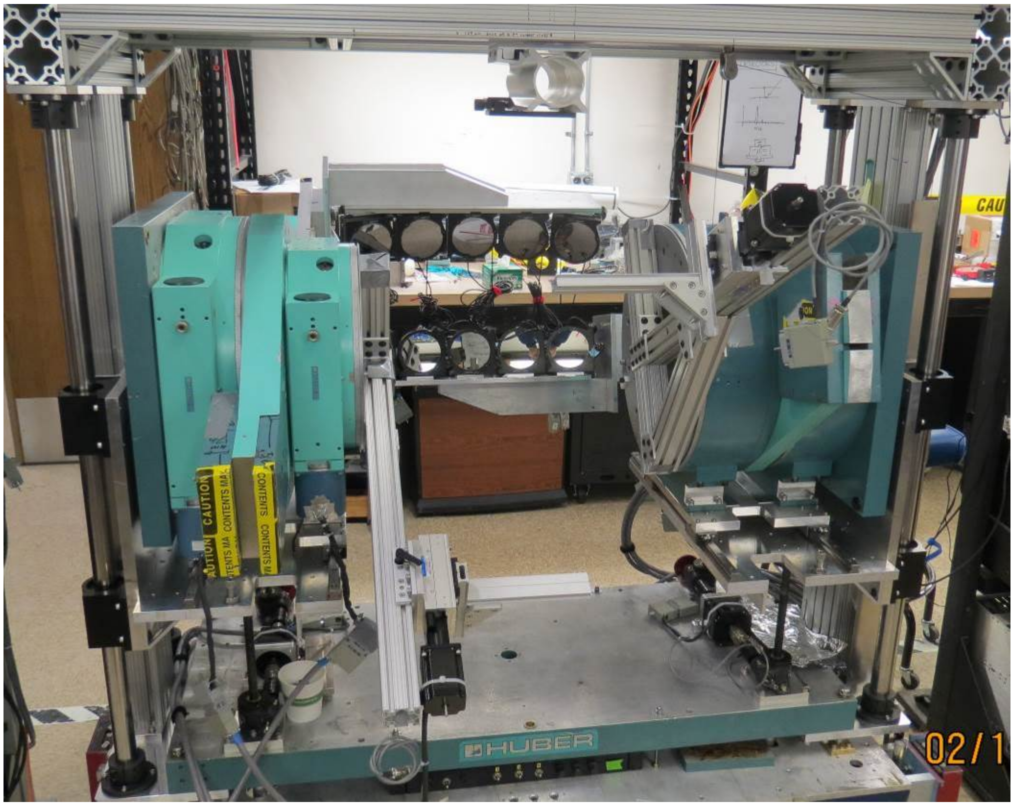
CHESS has developed and successfully deployed a novel Dual Array Valence Emission Spectrometer (DAVES) for high energy resolution, hard x-ray spectroscopy. DAVES employs the simplest method for scanning multiple spherically shaped crystals along a Roland circle. This new design achieves a unique 2-color collection capability and is built to take special advantage of pixel array detectors that significantly improve data quality when compared to existing systems. This presentation emphasizes the flexibility of experimental design offered by DAVES. Significant design principles are reviewed and several important applications are described and illustrated with results from DAVES first experimental testing period. Prospects and benefits of 2-color spectroscopy are discussed and future applications are considered.



The Dual Array Valence Emission Spectrometer (DAVES) has unique experiment flexibility, including simultaneous, independent collection of two-colors from one sample with doubled collection solid angle.

Ultra High Energy Resolution Focusing Monochromator for Inelastic X-ray Scattering Spectrometers*

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To bridge the gap currently existing between various spectroscopic techniques, Inelastic X-ray Scattering (IXS) spectrometers require X-ray optics with energy resolutions better than 0.1 meV and momentum resolutions better than 0.1 nm^{-1}. A concept of a spectrometer based on the backscattering geometry and exploiting dispersive properties of grazing incidence Bragg diffraction [1] can theoretically achieve the desired resolution but has a very narrow energy scanning range and a limited number of backscattering reflections. In our work, we propose a focusing monochromator combining focusing X-ray optics with conventional, i.e. non backscattering reflections, medium resolution monochromators that makes it possible to significantly improve the energy resolution otherwise limited by the crystal optics. The focusing monochromator can have a wide energy scanning range and can be designed for virtually any energy. Theoretical analysis of several optical layouts was supported by numerical simulations performed in "Synchrotron Radiation Workshop" software package using physical-optics approach and careful modeling of partially-coherent synchrotron (undulator) radiation. Along with the resolution, the shape of the energy resolution function was investigated. It was shown that under certain conditions the shape can be very close to Gaussian, which improves substantially the contrast of the energy-loss spectrum. The 0.1-meV or better energy resolution and the Gaussian shape of the energy resolution function make the focusing monochromator an ideal instrument for the IXS spectrometers.

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High-Resolution Soft X-ray RIXS Using Active Gratings and Energy Compensation

D. J. Huang, H. Y. Huang, W. B. Wu, H. S. Fung, J. Okamoto, H. W. Fu, S. W. Lin, C. C. Chiu, D. J. Wang, L. J. Huang, T. C. Tseng, S. C. Chung, and C. T. Chen

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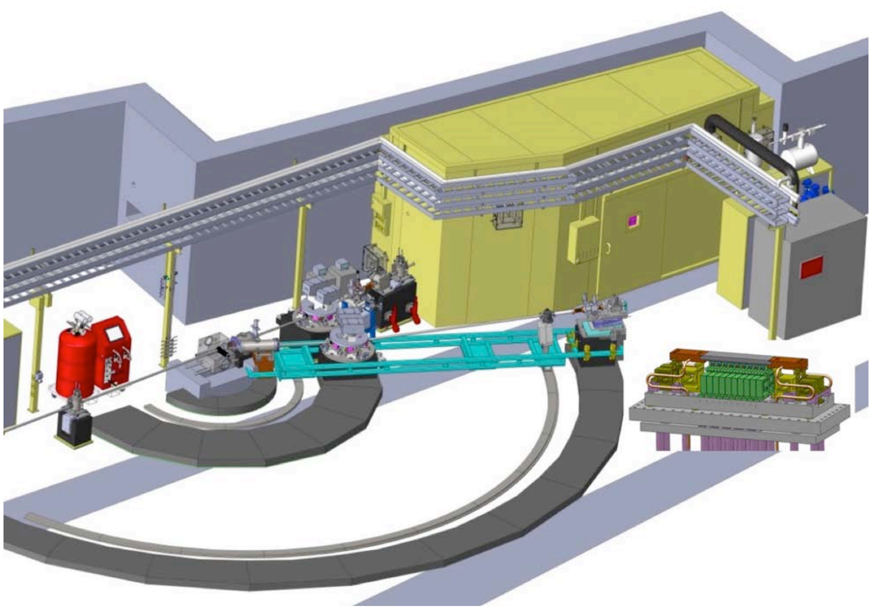
We have developed a unique technique for the high-efficiency and high-resolution beamline and spectrometer of inelastic soft X-ray scattering (RIXS). This new technique is based on the energy-compensation principle of grating dispersion. The design of the monochromator–spectrometer system greatly enhances the measurement efficiency at least by one order of magnitude. The setup comprises two bendable gratings to effectively diminish the defocus and coma aberrations. A test RIXS beamline of this design has been constructed at Taiwan Light Source, showing total energy resolutions of 65 meV and 130 meV at 710 eV and 930 eV, respectively [1]. This test beamline has yielded successful RIXS experiments of cuprate superconductors [2]. A new RIXS beamline based on this design will be established at Taiwan Photon Source. To reduce the grating surface deformation, a special grating bender is designed by adopting a multipoint scheme. A CCD detector with a sub-pixel spatial resolution through a centroid algorithm will be used. Our simulations indicate that the expected energy resolving power is better than 66000 at 530 eV and 45000 at 900 eV, respectively, with an efficiency one order of magnitude better than that of a conventional design.



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Development of a Novel Resonant Inelastic X-ray Scattering Spectrometer with Resolution Better than 10 meV

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We introduce the principles, design, and modeling of a novel resonant inelastic X-ray scattering (RIXS) spectrometer with resolution better than 10 meV, which is one order better than the typical energy resolution (~100 meV) of current RIXS spectroscopy. The main components of the new spectrometer include an inline nested channel-cut monochromator, a pair of focusing KB mirrors, and an analyzer system consisting of a Montel multilayer collimating mirror and a collimation-dispersion-selection (CDS) silicon-crystal-diffraction analyzer (see Fig. 1). The development of such a spectrometer at E = 11.215 keV is currently underway at the Advanced Photon Source as a strategic LDRD project. We will report the detailed development progress of this project, including the detailed parameters of the spectrometer system and fabrication and testing of the crystals and Motel mirrors. We believe that this new RIXS spectrometer, once fully developed, will revolutionize RIXS techniques, opening a window to a wide range of correlated electron systems currently inaccessible to RIXS. It will allow us to understand magnetic excitation spectra, electronic excitations, and electron-lattice coupling, for which theories and modeling constructs are in their infancy.

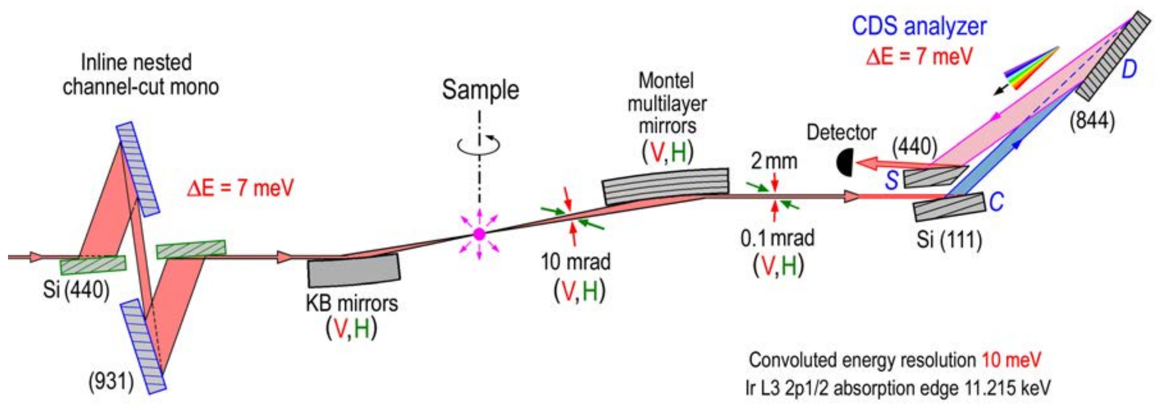

Fig. 1. Basic components and layout of the 10 meV RIXS spectrometer.

Measuring the Pathways to Complex Matter Far-From-Equilibrium: Development of Synchrotron X-ray Spatiotemporal Tools*

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From hard condensed matter to soft biological matter, every manifestation of nature exhibits far-from-equilibrium pathways leading to increasingly complex structural orders. A far-from-equilibrium state develops over time into organizations with length scales between atoms and small molecules on the one hand and mesoscopic matter on the other. Over the past century, almost all scientific effort has been to understand equilibrium structural order. However there is more focus in recent years on non-equilibrium physical, chemical and biological orders that demonstrate unique emergent properties. Examples include glassy metal alloys with unusual mechanical strength, nanomachines, emergent hidden correlations such as colossal magnetoresistance behavior, and mesoscale self-assembly of crystalline hybrid nanoparticles.

It is argued that progress in the understanding these complex order can only be made from a complete comprehension of the spatiotemporal evolution of non-equilibrium processes. How does one measure the evolution of complex order? Can we control the evolution? During the past decade many new experimental tools have been developed to modify the natural pulse structure of X-rays from storage-ring sources and XFELs in order to probe the evolution of electronic and chemical structure and properties over atomic to micron length scales and over fs to ms temporal scales. In this overview talk we will briefly summarize the successful progress made by the mechanical choppers in modifying natural time structure of the X-ray pulses from storage ring sources. The rest of the talk will focus on the recent development of X-ray photonic microsystems as a dynamic diffractive optics to generate nanosecond time-windows with over 100kHz repetition rates.[1,2] Further development of these systems with unprecedented design flexibility can lead to miniature X-ray optics with focusing and exceptional dynamic capabilities for wave-front manipulation, multicolor dispersion, and pulse slicing.[1]

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Fri

Plenary

8:30 AM

Synchrotron Research using Soft X-ray Resonant Inelastic Scattering

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Research using soft X-ray resonant inelastic X-ray scattering (RIXS) techniques has been developed at the ESRF for more than 20 years. Although, this has involved several groups, many of the developments have been possible due to a long standing collaboration with the group of L. Braicovich and G. Ghiringhelli at the Politecnico di Milano in Italy which has resulted in a steady evolution of the instrumentation at the ESRF and, directly and indirectly, in other facilities.

The scientific challenges, particularly in the area of correlated materials, like the cuprates, have spurred these improvements in instrumentation. In this talk, these challenges and the resulting instrumental developments will be discussed. The key parameter has been the improvement of the total energy resolution, which results from the joint optimization of the beam line (source, monochromator, refocusing) and of the spectrometer (sample manipulation, optical lay-out, grating, detector). This is the driving reason for a new instrument just being commissioned at the RIXS branch of the new ID32 beam line of the ESRF [1]. The beamline and spectrometer will be illustrated together with preliminary results, which indicate that the day-one expected energy resolution has already been reached.

Finally, since the ESRF instrument is just one of several that are being developed World-wide, the prospects for research using this powerful tool, in the coming years, will be discussed.

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Implications of Adding the Dimension of Time and Stimulated Processes to Science with X-rays

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Over the last years we have witnessed how X-ray pulse duration has become short enough to follow the kinetics and dynamics of functional materials or chemical reactions [1]. On these ultrafast time scales also the scattering duration time of resonant X-ray matter interaction, i.e. the natural core-hole life time, becomes comparable to the duration of intense pulses. Thus in a logical step we can transpose the powerful approaches of non-linear light-matter interaction along the historic line from NMR in the 1940`s via non-linear IR/VIS/UV laser spectroscopy to multi-photon and non-linear X-ray matter interaction [2,3,4]. I will discuss the implications of these developments for experimental investigations in general and review recent experimental findings.

Fri

Plenary

9:30 AM

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Real-time Data-intensive Computing

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Users don't seek out synchrotrons just as light sources or even just as data sources: increasingly, they want sources of understanding and discovery. The Advanced Light Source (ALS) has partnered with networking, high performance computing, and applied mathematics groups to create a "super-facility", giving users simultaneous access to both the experimental, computational, and algorithmic resources to make this possible. This combination forms an efficient closed loop, where data—despite its high rate and volume—is transferred and processed in real-time on appropriate compute resources, and results are extracted, visualized, and presented to users or to the experimental control system, both to provide immediate insight and to guide decisions about subsequent experiments during beamtime. We will describe our work at ALS ptychography, scattering, and tomography beamlines.

Propagation of Partially Coherent Beam through Non-ideal Beamline Components

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With the advent of new and planned synchrotron radiation sources with a high degree of coherence, accurate and efficient simulation tools for describing beam from partially coherent sources and its propagation through non-ideal beamline components are in high demand. At the APS, we have been exploring different theories and simulation methods to this end. We will present a fast beamline optimization tool based on a hybrid method combining ray tracing and wavefront propagation [1-3]. The hybrid method computes diffraction effects when the beam is clipped by an aperture or a finite optical acceptance and can also simulate the effect of imperfections (e.g., mirror figure errors) in the optical elements when diffraction is present. The partial coherence of the source is dealt with by the convolution method in order to improve the calculation speed. The method has been implemented in the ray tracing code SHADOW and benchmarked against other existing software (e.g., SRW). The results demonstrated that the hybrid code is advantageous in terms of its high efficiency and it is reasonably accurate for the purpose of beamline design and optimization. The recent improvements on the hybrid method and ideas for its future development will be discussed.

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Virtual Beamline Meets CHX Commissioning

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We have applied fully- and partially-coherent synchrotron radiation wavefront propagation simulation functions, implemented in the "Synchrotron Radiation Workshop" (SRW) computer code [1], to create a 'virtual beamline', mimicking the Coherent Hard X-ray (CHX) scattering beamline [2] currently undergoing commissioning at NSLS-II. The beamline simulation includes all optical beamline components, including insertion device, mirror with metrology data, slits, double crystal monochromator and refractive focusing elements. A specialty of the CHX beamline is the exploitation of X-ray beam coherence, boosted by the low-emittance NSLS-II storage-ring, for techniques such as X-ray Photon Correlation Spectroscopy (XPCS) or Coherent Diffraction Imaging (CDI). The key performance parameters are the degree of X-ray beam coherence and photon flux, and the trade-off between them needs to guide the beamline settings for the specific experimental requirements. The beamline simulations can not only be used to track the mutual intensity (Figure 1) after each optical element all the way to the sample position, but it can also be applied to simulate the coherent scattering pattern from a sample all the way onto the detector. The mutual intensity contains information about the degree of transverse coherence, which can be measured e.g. from slit diffraction. Moreover, common characterization experiments for the coherence of the X-ray, such as scattering from a Boron fiber, can be directly simulated [3] and provide thus valuable information about the performance of optical elements.

Simulations of key performance parameters will be compared to early commissioning results, including degree of transverse coherence, flux, simulated and measured coherent scattering patterns.

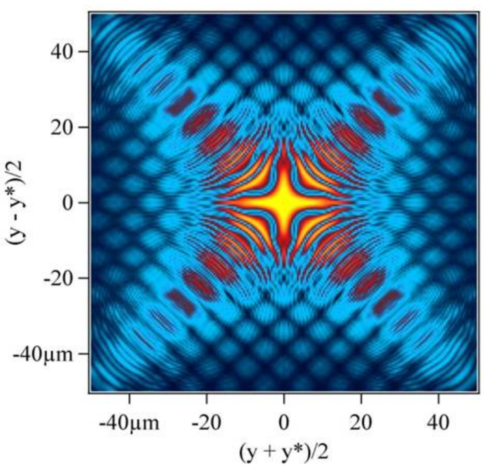

Figure 1. Vertical median plane of the simulated mutual intensity at the sample position of the CHX beamline, using settings for high transverse coherence.

The work was supported by US DOE grant No. DE-SC0011237.

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A Novel Optical Design for a Micro-focusing Beamline

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A novel optical setup has been designed for a micro-crystallography beamline at SSRF, by using a convex mirror to obtain a virtual focus and then to focus the beam to a few microns by a concave mirror. In this way, we achieved a larger demagnification ratio for the horizontal beam size comparing with K-B mirrors. In practice, we used an anticlastic mirror to focus the beam vertically and to defocus the beam horizontally, and together with one cylindrical mirror to focus the beam horizontally. Shown in figure 1 is the beamline optical setup. The source of beamline is an in-vacuum undulator of 25 mm period length and the size of source is about 380×23μm^2. The distance from source to sample position is 45 meters and the center of last mirror to sample position is 1.7m, which gives more than one meter long space for beam tailoring and experimental instruments. The beamline has been completed and the optical system has shown a good performance with a minimum focused beam size of 7.5×5μm^2 obtained. The demagnification ratio on the focused beam size is about 51:1, which is about twice of that the K-B mirrors can achieve. This focusing optical setup has shown two unique features: the demagnification ratio can be easily adjusted with fixed working distance and very large demagnification ratio can be achieved, which is only limited by the minimum curvature of mirrors.

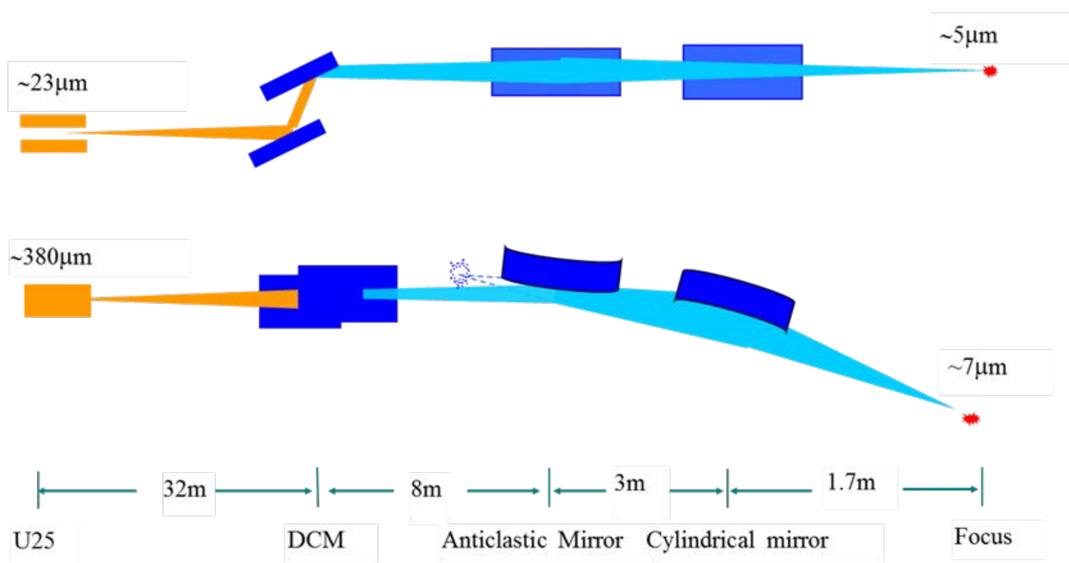

Figure 1. Layout of beamline optical setup

Synchrotron Infrared Beamline Design

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Synchrotron infrared beamlines are powerful tools to perform spectroscopy on microscopic length scales but require working with high intensities which are obtained using large horizontal beamline apertures. Most of the infrared beamlines use a single toroid shaped mirror to conjointly focus both the vertical and the horizontal source emission but generate for large apertures distorted images due to the optical aberrations produced by the depth and the circular shape of the source. In this presentation, we will describe a new optical layout consisting in two optimized shape mirrors, focusing respectively the vertical and the horizontal source emission, and providing low aberration beams for large horizontal apertures. This layout is already operational on the IR beamline of the LNLS /1/.

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An Accurate Optical Design Method for Synchrotron Radiation Beamline with Wave-front Aberration Theory

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For synchrotron beamline design, aberrations present within the optics have been treated successfully by theories which relate the aberration coefficients with Taylor expansion coefficients of the light path function [1, 2]. Nevertheless, the applied scope has some limitation, as pointed out by Lu et al. [7], high order aberration items are not accurate and the spectral aberration usually is not equal to linear sum of aberration items in the light path function [3]. Nowadays, the spectral resolution of beamlines is extremely high, with the sizes of the source and image points in the order of microns. Calculation of aberrations with a high degree of accuracy is therefore very crucial for beamline performance, especially for a double-element monochromator. Namioka used a procedure known as the SD method [4,5] (the analytical formulae of a ray-tracing spot diagram), to find the optimal optical parameters using a statistical process to minimize the aberrations. This was successful to some extent, but it is quite complicated and although this method is accurate, there is no way to understand how the aberration terms are individually cancelled, with residual aberrations remaining without being handled. In this paper, we present a method to calculate the aberrations of a double-element monochromator with a high degree of accuracy. This is based on Lu's wave-front aberration method [7,8], developed using Chrisp's methodology [4], to demonstrate successfully the treatment of multiple aberrations of optics in an intuitive manner. The results are compared to a design example with the SD method [9] and the light path function method [1,2]. Our method clearly shows the physical contribution of the aberrations, and the way in which to minimize them becomes straightforward.

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RAY-UI: A Powerful and Extensible User Interface for *RAY*

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The RAY-UI project started as a proof-of-concept for an interactive and graphical user interface (UI) for the well-known ray tracing software RAY [1]. In the meantime, it has developed into a powerful enhanced version of RAY that will serve as the platform for future development and improvement of the source code and associated tools.

The software as of today supports nearly all sophisticated simulation features of RAY. Furthermore, it delivers very significant usability and work efficiency improvements. Beamline elements can be quickly added or removed in the interactive *sequence view*. Parameters of any selected element can be accessed directly and in arbitrary order. With a single click, parameter changes can be tested and new simulation results can be obtained. All analysis results can be explored interactively right after the ray tracing by means of powerful integrated image viewing and graphing tools. Unlimited image planes can be positioned anywhere in the beamline, and bundles of images planes can be created for moving the plane along the beam to identify the focus position with live updates of the simulated results.

In addition to showing the features and workflow of RAY-UI, we will give an overview of the underlying software architecture as well as examples for use and an outlook for future developments.

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Capturing Structural Dynamics of Photocatalyst by Picosecond X-ray Pulses

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Picosecond time-resolved X-ray techniques, such as time-resolved X-ray diffraction, scattering, and spectroscopy, utilize the pulsed nature of synchrotron radiation from storage rings, and are becoming general and powerful tools to explore structural dynamics in various materials. This method enables to produce "atomic structural movies" at picosecond temporal resolution. It will be fascinating to apply such capability to capture ultrafast structural dynamics in advanced materials of strongly-correlated electron systems, photochemical catalytic reaction dynamics in liquid or on solid surface, light-induced response of photosensitive proteins, etc.

For example, ultrafast X-ray spectroscopy offers new opportunities for elucidating ultrafast structural dynamics both in solid and solution. X-ray absorption fine structure (XAFS) spectroscopy reveals details about the local geometric and electronic structure around selected atoms. The extended X-ray absorption fine structure (EXAFS) provides accurate bond distances, while the X-ray absorption near-edge spectroscopy (XANES) delivers additional information about unoccupied orbitals and spin configurations [1-6].

Photon Factory Advanced Ring (PF-AR) at the High Energy Accelerator Research Organization (KEK), Tsukuba, Japan is a 6.5-GeV electron storage ring dedicated for single-bunch operation and is suitable for the picosecond time-resolved X-ray studies. An in-vacuum undulator beamline NW14A at the PF-AR was designed and constructed to conduct a wide variety of time-resolved X-ray measurements, such as time-resolved X-ray diffraction, scattering and spectroscopy. Successful examples of time-resolved XAFS studies applied to photocatalyst will be presented.

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THz Coherent Synchrotron Radiation used for Ultra High Resolution Spectroscopy and Ultra-fast THz Measurements

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During the last decade, an unprecedented high power source of THz radiation was made scientifically available: coherent synchrotron radiation (CSR) [1-3]. This radiation produced from relativistic electron bunches of picosecond duration opens up new territory in the THz range with intensities up to 4 orders of magnitude higher than previous sources. In this mode of operation (200 electron bunches of 100 μA each), we showed that the stability is sufficient to perform bunch per bunch measurements based on Electro Optics sampling technique.

Other extreme properties have been recently unveiled. In particular, the CSR emission forms two frequency combs (FC) generated over one decade of frequency. The first one originating from the electron bunch to bunch repetition is the signature of a degree of coherence amongst the bunches and is designated as super radiance emission [5]. Recent heterodyne mixing measurements reveal a second FC composed of sharp teeth regularly spaced by 846 kHz and related to the revolution period of the electron bunches in the storage ring (1.18 μs). It produces a spectrally dense THz FC with more than 10^6 components covering the THz range from 0.1 to 1 THz. This FC presents unprecedented properties such as high power, broad frequency range, zero frequency offset, and high density. These properties allow the CSR FC to be exploited for ultra-high resolution spectroscopy.

The CSR discrete emission reveals that for the THz coherent emission, the entire ring behaves in a similar fashion to a resonator wherein electron bunches emit pulses quasi-synchronously.

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Time-Resolved X-ray Spectroscopy @ P04 of PETRA III

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Most chemical and all biological processes are occurring in a liquid environment. Yet, is an experimental challenge to map the electronic configuration since the K-edges of life important elements, e.g. C, N, O are in the soft X-ray regime. It becomes even more difficult to follow the dynamics of these systems with a sufficient temporal resolution on the atomic level. The time resolved experimental approach allows to elucidate the understanding the origin of molecular motions, e.g. isomerization, dimerization, charge transfers etc. in real time.

Here, we would like to present a next generation experimental endstation, TrRIXS, for time-resolved soft X-ray spectroscopy performed on liquids on a pump-probe scheme at soft X-ray beamline P04 of the synchrotron storage ring PETRA III, see figure 1. So far, time-resolved pump-probe soft X-ray spectroscopic studies have been performed mainly at free electron lasers due to higher photon intensities and equal native repetition rates of pump and probe beam. In presented set-up, the laser pump and X-ray probe synchronization is based on an extended PLL approach with two interconnected feedback loops and a timing jitter of lower than 1 ps rms. As a detection device, a newly soft X-ray spectrometer based on reflection zone plate design has been developed, which provide high energy resolution with high photon detection efficiency enabling to perform X-ray emission and X-ray absorption measruements. The endstation presented here opens up new possibilities to study dynamics of chemical and biological systems in liquid environment on the atomic scale.

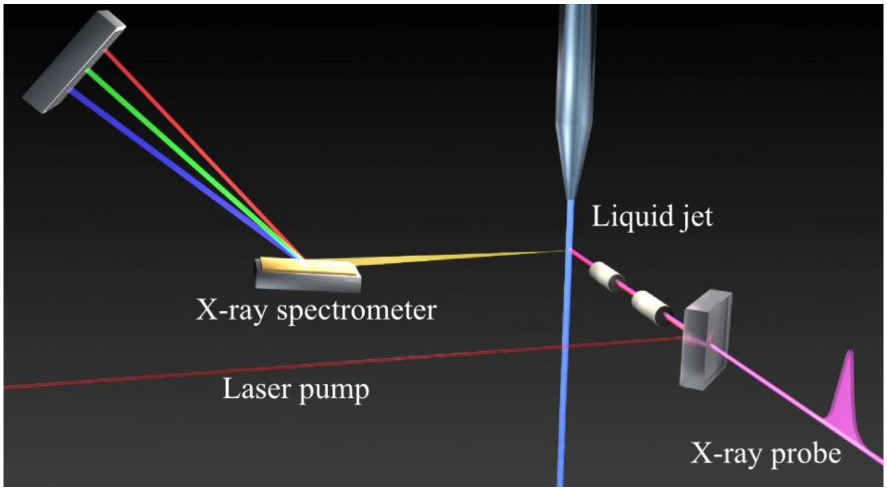

Figure 1: Scheme of the experimental pump-probe set-up.

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Ultrafast X-ray Diffraction at High Repetition Rates at the XPP-station at BESSY II

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Since April 2015, the new XPP-KMC3-Beamline at the synchrotron source BESSY II is operational and available to external users. The XPP experimental station is dedicated to ultrafast X-ray diffraction at high repetition rates and aims at studying ultrafast lattice dynamics in solids and nanostructures. A core element of the experiment is the ultrafast fibre based pulsed laser system. The laser system allows for sample excitation with 250 fs optical pulses at repetition rates up to 1.25 MHz, the repetition rate of the storage ring.[1] The laser is electronically synchronized to the X-ray pulses from the synchrotron, thus allowing for optical pump – X-ray probe experiments. A second core element is the 4-circle diffractometer which is mounted in a vacuum vessel for sample cooling down to less than 50 K.

We will present a detailed description of the experimental setup. The specifications and parameters will be discussed by presenting first experimental results obtained with the new setup. First measurements were performed on ultrafast phase transitions, nanoscale thermal diffusion[2] and coherent phonon dynamics in thin films[3]. We will lay out the scientific scope of the beamline by discussing possible future applications of our new setup. Finally, we present new results on the development of an ultrafast X-ray Bragg switch which aims at shortening the synchrotron X-ray pulse from 100 ps to less than 5 ps.[4]

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Dynamics of Charge-ordering in Superconducting Cuprates Studied using Time-resolved Resonant Soft X-ray Diffraction

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The effects of charge, spin and structural ordering on superconductivity in High-T_c copper oxides remains one of the great mysteries of condensed matter physics. For instance, in the archetypical superconductor $La_{2-x}Ba_xCuO_4$ (x=1/8) charge ordering along with a low temperature tetragonal (LTT) lattice distortion appear at the same temperature (~55K). Importantly, the superconducting transition temperature is greatly suppressed for x=1/8 doping, but the mechanism for this suppression remains largely a matter of debate. In order to gain insights into this problem we have used Time-Resolved Resonant Soft X-Ray Diffraction (TR-RSXD), employing a pump-probe approach combining optical excitation with an x-ray probe, to disentangle the competing effects of charge-ordering and LTT distortions in the high-T_c superconductor $La_{2-x}Ba_xCuO_4$. The experiments were performed at Diamond Light Source (UK) and the Linac Coherent Light Source (USA). The TR-RSXD results show that photoexcitation using near-IR (800nm) or mid-IR (14.5μm) wavelengths creates a transient phase where charge-ordering is destroyed while the LTT structural distortions remain largely intact [1,2]. Simultaneously, transient enhancements of superconductivity along the *c*-axis following the destruction of charge-ordering were observed in time-resolved THz probe experiments. The results, as a whole, imply that around the 1/8-doping level charge-ordering alone prevents superconductivity in the High-Tc materials.

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Synchrotron VUV Photoionization Mass Spectrometry and its Applications on the Analysis of Pyrolysis Products of Solid Materials in Real Time

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Recently, synchrotron vacuum ultraviolet (VUV) photoionization mass spectrometry (SVUV-PI-MS) has been applied for the online analysis of the chemical components and their highly dynamic processes during pyrolysis and combustion of various fuels. Compared to traditional "hard" electron ionization (EI) methods for the gaseous components analysis, photoionization produces little or no fragments, making the identification and interpretation of complex ingredients in real-time possible.

In this work, a newly constructed SVUV-PI-MS setup at the mass spectrometric analysis endstation of National Synchrotron Radiation Laboratory (NSRL) of China will be introduced, which is mainly designed for the pyrolysis/combustion studies of solid materials[1-3]. The applications of our SVUV-PI-MS are outlined in two aspects: (1) Online analysis for the pyrolysis products of municipal waste polymers, such as polyethylene (PE) and polypropylene (PP). Mass spectra of the products at different temperatures (400-900 °C) were measured during the pyrolysis process. Time-evolved profiles of pyrolysis products of PE and PP were also recorded with SVUV-PI-MS. In addition, catalyst effects could be observed in real time; (2) Online study on the combustion of biomass, cigarettes, and artemisia argyi, a traditional Chinese medicine. By virtue of the high tunability of synchrotron radiation, smoke components during combustion can be qualitatively determined by their different IEs, even isomers can be distinguished by measuring photoionization efficiency (PIE) spectrum. In addition, mass spectra of smoke components at different photon energies can be obtained in a few seconds without chromatographic separation. Thus, the dynamic processes of those nascent products can be studied in real time.

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Compressed Sensing and Processing for Rapid Three-Dimensional Nanoimaging

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The shift to dynamic three-dimensional imaging requires the development and implementation of advanced data processing methods and their close integration with the instrument. Unlike other methods, compressed sensing (CS)-based approaches are particularly well suited for nanoimaging applications. These methods rely on the sparse representation of the data and promote the use of optimized data collection strategies that can significantly reduce total x-ray exposure to samples. They also have great potential to decrease total acquisition time making the system less sensitive to any instrumental drifts. In this talk, we will describe an iterative CS-based computational framework and evaluate various data sampling schemes for nanoimaging applications. Similar to other iterative algorithms the proposed method requires a high performance computing infrastructure to timely handle the processing, storage and distribution of large datasets. We will present the processing workflow between the Advanced Photon Source and the Argonne Leadership Computing Facility supercomputers, and discuss key components and benefits for a coherent integration of acquisition and data parallelism for synchrotron experiments.

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GigaFROST: the Holy Grail of Fast Tomography.

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With the latest developments in CMOS technology, it has been possible to exploit tomographic microscopy at third generation synchrotron facilities to unprecedented levels [1,2]. However, one important limitation has remained: a generally short total acquisition time at high frame rates due to the limited internal memory of available detectors. To address and solve this flaw, we have developed GigaFROST (Fig.1), a new detection system based on a commercial CMOS sensor [3] acquiring and streaming continuously 7.7 GB/s of data directly to a server. The images are temporarily stored into the server's RAM and processing can be performed at this early stage before actually writing the data permanently to disk. This allows data pre-processing as well as data reduction, a more and more necessary step considering the amount of data acquired in a typical fast tomographic experiment at a synchrotron beamline (~2 Tb/day). We finally succeeded to decouple temporal resolution from total acquisition time – a sort of Holy Grail for fast imaging systems. We present first experimental data acquired with GigaFROST highlighting the capabilities of this new instrument set-up. We will show scientific cases that could only partially be addressed in the past but have become feasible with the introduction of GigaFROST with special emphasis on in situ and in vivo applications.

Fig.1 The Gigafrost detector

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Fast and Time-resolved Tomography at ANKA: Applications, Infrastructure and Data Management

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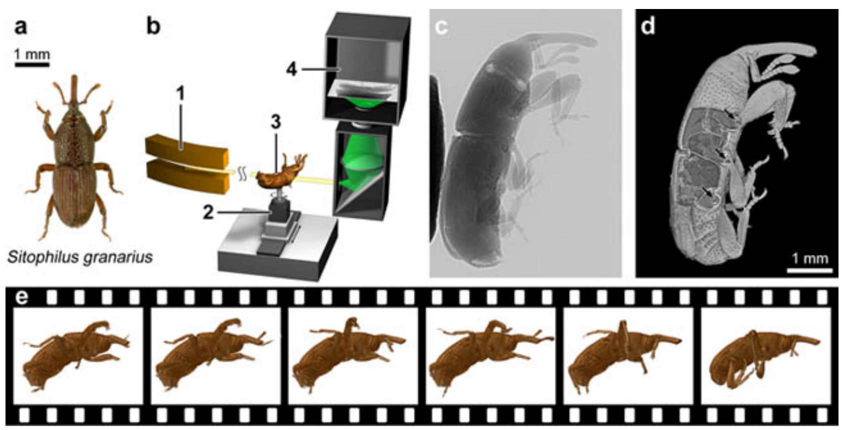

Fig. *In vivo* X-ray cine-tomography. (a) *S. granarius*, dorsal view. (b) Experimental setup showing bending magnet (1), rotation stage (2), fixed specimen (3), and detector system (4). (c) Radiographic projection. (d) Three-dimensional rendering of the reconstructed volume. (e) Cine-tomographic sequence of moving weevil.

The best method to understand internal movements in organisms is to observe their dynamics in 4D. Aiming to investigate fast movements with feature sizes in the micron range and with temporal resolution down to a few tens of milliseconds, we developed *in vivo* X-ray cine-tomography[1]. It comprises ultra-fast SR-μCT and 3D reconstruction, automated data processing and motion analysis procedures. We demonstrate the method by investigating the dynamics of screw-and-nut type hip joints[2] in granary weevils (*Sitophilus granarius*).

At ANKA, we enabled the ultra-fast tomography workflow in the German-Russian UFO project and the *Verbundforschung* project ASTOR. In UFO we developed intelligent system design, vast computational power and sophisticated algorithms[3]. On-line assessment of sample dynamics facilitates active image-based control. The new control system *Concert* seamlessly integrates computing nodes, a low-level camera interface and fast control[4].

Consequently, the recent progresses in high speed X-ray tomography resulted in a dramatically increase of data volumes. In the scope of ASTOR, we established a virtual analysis environment for large-scale datasets with tailored analysis methods for functional morphology.

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Ophyd: Software for Data Collection, Management, and Analysis

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Ophyd is a Python-based framework for experiment control under development at Brookhaven National Lab and deployed for use on NSLS-II Project beamlines. The software supports device control, data collection and distribution, reciprocal space operations, integration with electronic logging, and more. Ophyd is coupled with searchable back-end storage and retrieval components, which together form an end-to-end data management architecture for the NSLS-II. An overview of Ophyd's architecture, infrastructure, and features will be discussed.

A New Approach to Synchrotron Radiation CT Imaging and Powder Diffraction using Hard X-ray Detector and Fast Continuous Framing

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The Maxipix-CdTe photon-counting detector features a 1mm thick CdTe sensor hybridized to three Timepix chips, arranged in an array of 768x256 pixels at 55 µm pitch. Such sensor is 99% efficient up to 60 keV. Each pixel features an energy threshold, providing noise free images at room temperature. The readout dead time is 290 us [1]. The frame rate limitation is overcome using continuous scan techniques, where a frame is transferred while the next is acquired and readout. The following two examples require both a hard X-ray detector as well as continuous frame acquisitions.

The first experiment uses synchrotron radiation computed tomography for iodine contrast agent concentration measurements with monochromatic beam at 35 keV. Whereas the reference Germanium detector is a 1D strip detector, here 2D tomography projections are acquired at each angular position of the motor which is rotating at speeds as fast as 180 degrees per second (minimum acquisition time 3 ms). Continuous scan allows fast full 3D acquisitions, enabling low dose imaging. The iodine concentration is dynamically followed in time.

The detector has also been used in powder diffraction experiment at 40 keV. Unlike with the CCD camera, noise-free images are taken. To achieve data collection at very high Q values, the detector was scanned along the axis perpendicular to the beam. Total scan distance was 320 mm. Therefore using the continuous scan we were able to bypass the small detection area of the detector, and take statistically relevant data at high speed (3 ms per frame).

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General Method for Automatic on-line Beamline Optimization Based on Genetic Algorithm

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It is essential but inconvenient to perform high quality on-line optimization for the synchrotron radiation (SR) beamline. Usually, SR beamlines are optimized manually, which is time-consuming and difficult to obtain global optimization for all optical elements of the beamline. We developed a general method for automatic beamline optimization based on the Genetic Algorithm [1]. This method can optimize all optical components of any kind of beamline simultaneously and efficiently. A program developed using Labview is examined at XAFCA [2] beamline to optimize the beam flux at the sample position. The experimental results demonstrate that the beamline can be optimized within 20 generations even when the initial flux is as low as 4% of its maximum value

GA is a computational analogy, simulating the main process of natural selection, to solve optimization and search problems. As a global optimization algorithm, it can be applied to various systems, such as synchrotron radiation beamlines, which are difficult to formalise mathematically. This method only requires the input and its output of the objective system and does not require the mathematical model between them. Therefore, it is independent of a particular beamline set up and can be expanded further to other beamlines and scientific instrumentations.

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Synchrotron Infrared Nano-Spectroscopy

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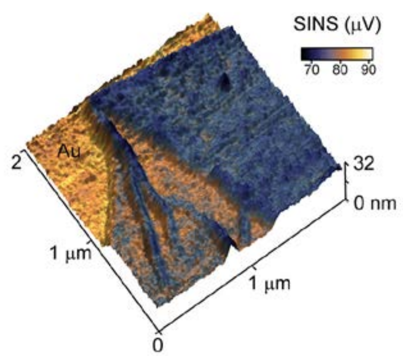
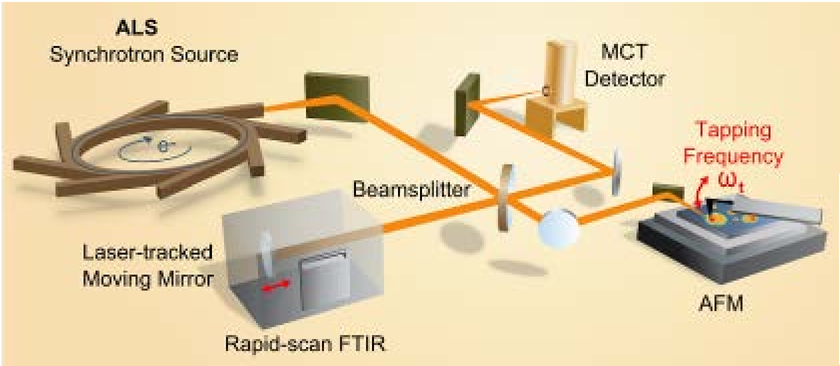
By combining scattering-scanning near-field optical microscopy (s-SNOM) with mid-infrared synchrotron radiation, synchrotron infrared nano-spectroscopy (SINS) enables molecular and phonon vibrational spectroscopic imaging, with rapid spectral acquisition, spanning the full mid-infrared (500-5000 cm^{-1}) region with nanoscale spatial resolution. This highly powerful combination provides access to a qualitatively new form of nano-chemometric analysis with the investigation of nanoscale, mesoscale, and surface phenomena that were previously impossible to study with IR techniques. We have installed a SINS end-station at Beamline 5.4 at the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory, making the s-SNOM technique widely available to non-experts, such that it can be broadly applied to biological, surface chemistry, materials, or environmental science problems. We demonstrate the performance of synchrotron infrared nano-spectroscopy (SINS) on semiconductor, biomineral and protein nanostructures, providing vibrational chemical imaging with sub-zeptomole sensitivity.



This peptoid nanosheet, produced by Gloria Olivier and Ron Zuckerman at Berkeley Lab, is less than 8 nanometers thick at points. SINS makes it possible to acquire spectroscopic images of these ultra-thin nanosheets for the first time.

This talk will present the novel SINS instrumentation and a variety of scientific examples. Future directions, both technical and scientific, will be discussed.



Experimental setup for SINS that includes the synchrotron light source, an atomic force microscope, a rapid-scan Fourier transform infrared spectrometer, a beamsplitter, mirrors and a detector.

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Ultrahigh Energy Resolution is Achieved in Dreamline at Shanghai Synchrotron Radiation Facility

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A new ultrahigh-energy-resolution and wide-energy-range soft X-ray beamline (named Dreamline) has been constructed at the Shanghai Synchrotron Radiation Facility. The beamline construction was started in January 2010 and finished in October 2014. The energy resolving power is achieved to be 50,000 ($E/\Delta E$) @867eV, which is the best value in the world. The flux at this energy resolution is 1.2E12 phs/s. The energy resolution of the angle-resolved photoemission spectroscopy (ARPES) is 3.7meV with Helium lamp. The spatial resolution in the photoelectron emission microscopy (PEEM) is 17nm @200eV.

The beamline has two branches: one dedicated to ARPES and the other to PEEM. The two branches share the same plane-grating monochromator, which is equipped with four variable-line-spacing gratings and covers the 20–2000 eV energy range. Two elliptically polarized undulators are employed to provide photons with variable polarization, linear in every inclination and circular, and different energy range, 20-200eV and 200-2000eV, respectively. The refocusing of both branches is based on Kirkpatrick–Baez pairs. The use of plane optical elements upstream of the exit slit, a variable-line-spacing grating and a premirror in the monochromator that allows the influence of the thermal deformation to be eliminated are essential for achieving the ultrahigh-energy resolution. The monochromator and exit slit are thermal controlled at ±0.1°C to realize an energy stability of <10meV during 24 hours.

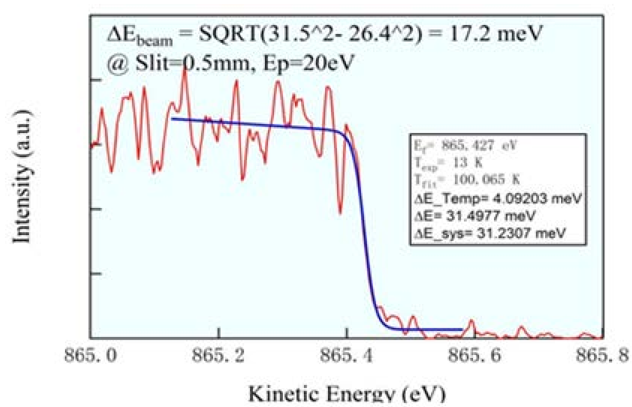

Fig. 1. Energy resolution measurement with ARPES spectrum of copper

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High- field THz from 4th Generation Light Sources: THz Beamline at FLASH

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Last decade witnessed the rise of new type of accelerator light sources based on linear accelerators. Like synchrotron sources, these large-scale facilities have the appeal of their broad spectral range (from THz to X-rays) as well as tunability. However, their advantage over synchrotrons lays in the ability to generate ultrashort pulses with peak intensities many orders of magnitude higher.
In THz range, the ability of 4th generation sources to generate pulses with electric field strength in the GV/m range, enabled number of exciting experiments, especially in the field of non-linear THz spectroscopy and THz control experiments. A variety of genesis concepts allows shaping the THz pulses from single-cycle (broadband) pulses to many-cycle narrow-bandwidth pulses and polarizations ranging from radial to linear. The main advantage of accelerator-based THz originates from the fact that the THz generation process does not take place in a medium but in the accelerator vacuum, and therefore the THz pulse energy can be scaled up much easier than in case of the table top sources available today. In addition, it has been demonstrated recently that coherent THz radiation can be generated along femtosecond X-ray pulses in 4th Generation X-ray Light sources such as FLASH [1, 2, 3] and LCLS [4]. This opens up the exciting opportunities for naturally synchronized THz pump X-ray probe experiments on few femtosecond time scales [1, 2, 4]. We present an overview of high-field THz facility at FLASH/DESY and experimental opportunities and challenges are discussed with the example of recent experiments.

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Electronic Ordering States in Strongly Correlated Electron Systems Studied by Resonant Soft x-ray Scattering

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Various interesting physical properties, such as colossal magnetoresistance effect and magnetoelectric effect, have been discovered in *3d* transition metal compounds. There the strong coupling among charge, spin, and orbital degrees of freedom play important roles. The study of these electronic ordering states is essential to understand the phenomena microscopically. Resonant soft x-ray scattering (RSXS) is quite effective technique to study the electronic orderings, since it can directly probe the *3d* electronic state utilizing the $2p \rightarrow 3d$ dipole process at the $L_{2,3}$-edge. Hence the RSXS measurements become to be performed globally. In the Photon Factory, the diffractometers were developed for the RSXS measurement, [1] and started to investigate the electronic ordering in the *3d* transition metal compounds. [2] In this talk, recent scientific topics will be presented.

One is the study of $LaCoO_3$ film, which shows unique ferri-magnetic ordering, although the ground state of the bulk $LaCoO_3$ is the non-magnetic state with the Co^{3+} low-spin state. We clarified the peculiar spin-state and the orbital order of Co^{3+} induced by the epitaxial strain effect. [3] Moreover, we investigated the surface state of electronic orders by surface sensitive grazing-incident RSXS. The transition temperatures of orbital and magnetic order were observed to be different between the surface and the bulk. Finally we revealed the non-trivial surface electronic state.

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Innovative Diffraction Gratings for High-resolution Resonant Inelastic Soft X-ray Scattering Spectroscopy

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High-resolution Resonance Inelastic X-ray Scattering (RIXS) requires diffraction gratings with very exacting characteristics. The gratings should provide both very high dispersion and high efficiency which are conflicting requirements and extremely challenging to satisfy in the soft x-ray region for traditional grazing incidence geometry. To achieve high dispersion one should increase groove density of a grating; this however results in a diffraction angle beyond the critical angle range and results in drastic efficiency loss. The problem can be solved by use of multilayer coated blazed gratings (MBG). Although these types of diffractive element have been studied experimentally and theoretically, up to this point there isn't a framework that allows optimization of the many parameters of the structure. In this work we have investigated the diffraction characteristics of MBGs via numerical simulations and have developed a procedure for optimization of grating design for particular applications. We apply the procedure for optimization of MBGs for a multiplexed high resolution imaging spectrometer for RIXS spectroscopy [1] to be built in sector 6 at the ALS. We found that highest diffraction can be achieved for the gratings optimized for the 3rd or 4th order operation. Fabrication of such gratings is an extremely challenging technological problem and requires innovative fabrication approaches. We have evaluated non-traditional grating fabrication techniques such as direct-write optical and e-beam lithography, nanoimprint lithography, wet anisotropic etch, and conformal multilayer growth. We will present the first experimental prototypes of these gratings and report their performance. High order – high line density gratings have the potential to be a revolutionary new optical element that should have great impact in the area of soft x-ray RIXS. The work was supported by the US Department of Energy under contract number DE-AC02-05CH11231.

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A New Generation of X-ray Spectrometry UHV Instruments at the SR Facilities BESSY II, ELETTRA and SOLEIL

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A novel type of ultra-high vacuum instrument for X-ray reflectometry and spectrometry related techniques for nanoanalytics by means of synchrotron radiation has been constructed and commissioned at BESSY II [1]. This versatile instrument was developed by the Physikalisch-Technische Bundesanstalt, Germany's national metrology institute, and includes a 9-axis manipulator that allows for an independent alignment of the samples with respect to all degrees of freedom. In addition, a rotational and translational movement of several photodiodes as well as a translational movement of a beam geometry defining aperture system is provided. Thus, the new instrument enables various analytical techniques based on energy dispersive X-ray detectors such as reference-free X-Ray Fluorescence (XRF) analysis, total-reflection XRF, grazing-incidence XRF, in addition to optional X-Ray Reflectometry (XRR) measurements or polarization-dependent X-ray absorption fine structure analyses. Samples having a size of up to 100 mm x 100 mm can be analyzed with respect to their mass deposition, elemental, spatial or species composition with respect to surface contamination, nanolayer composition and thickness, the depth profile of matrix elements or implants, nanoparticles or buried interfaces as well as the molecular orientation of bonds.

Three technology transfer projects of adapted instruments have enhanced X-Ray Spectrometry (XRS) research activities within Europe at the synchrotron radiation facilities ELETTRA (IAEA) and SOLEIL (CEA/LNE-LNHB) as well as at the X-ray innovation laboratory BLiX (TU Berlin) where different laboratory sources are used. Here, smaller chamber requirements led PTB in cooperation with TU Berlin to develop a modified instrument equipped with a 7-axis manipulator: reduced freedom in the choice of experimental geometry (absence of out-of-SR-plane and reference-free XRS options) has been compensated by encoder-enhanced angular accuracy for GIXRF and XRR. Selected applications of these advanced ultra-high vacuum instruments demonstrate its flexibility, capabilities and reliability.

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Progress of Nanopositioning Stages Development for Hard X-ray Nanofocusing and Coherence Preservation Optics at the APS

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With brighter upgraded APS diffraction-limited x-ray sources and more advanced x-ray optics, scientists at the APS will need more customized novel x-ray instruments. Nanopositioning techniques present a significant opportunity to support the state-of-the-art synchrotron radiation instrumentation research for the APS upgrade project. Precision ball-bearing- or roller-bearing-based positioning stage systems provide large travel range. However, it is not possible to meet requirements in sub-nanometer positioning resolution, high tilting stiffness, and sub-microradian straightness of trajectory repeatability with a single guiding system. Customized flexure mechanisms and precision thermal expansion compensation are needed for the development of nanopositioning stages for hard x-ray nanofocusing and coherence preservation optics at the APS. Recent progress of such stages development is presented in this paper, which includes stages designed for:

- Alignment apparatus for K-B mirrors with 20 - 50 nm focal spot [1];
- Alignment apparatus for six Fresnel zone plates stacking with 20 nm focal spot (see figure 1) [2];
- Wire scan device for 3-D x-ray diffraction microscope differential aperture [3];
- UHV hard x-ray monochromators for coherence related applications [4]; and
- Four-crystal hard x-ray split-and-delay line with coherence preservation [5].

Preliminary test results for mechanical performance of these nanopositioning stages are also discussed in this paper. This work is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Contract No. DE-AC02-06CH11357.

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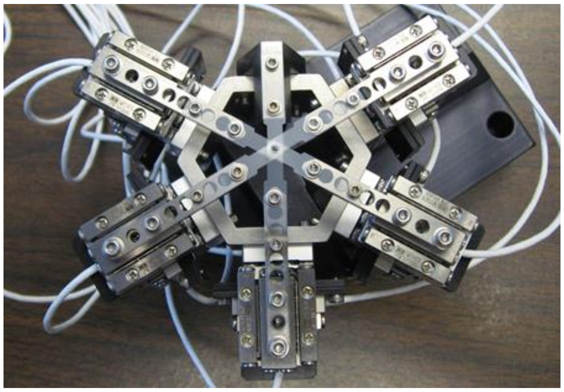

Figure 1, A potograph of the alignment apparatus for six Fresnel zone plates stacking with CVD diamond holders.

Mounting and Cooling Effects of X-ray Mirrors on the Nanometer Scale

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European XFEL and the new generation light sources will produce photon beam with unique characteristics: almost diffraction-limited, fully coherent, ultra-short pulses and MHz repetition rate. In order to preserve the beam properties and also to cope with the optics damage issue the photon transport systems are hundreds of meters long and foresee optical elements up to 1 meter long, cooled and with outstanding quality of the reflecting surface (below 100 nrad slope error and the peak-to-valley residual height error in the nanometer range). Those mirror specifications represent a challenge under manufacturing, measuring and engineering point of view. One of the main engineering challenges is to maximize the mechanical and thermal stability. Position, number, material and design of the supports are sensitive variables for the mirror stability and for the minimization of the optical surface perturbation. Moreover the outstanding quality surface should be preserved also under variable thermal load and therefore the substrate shape and the geometrical features on it have to be carefully optimized for the wide energy operational range. Status of development in this field at European XFEL is reported in this paper.

Optimizing X-ray Mirror Thermal Performance using Matched Profile Cooling

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High heat-load issues on X-ray optics continue to be a key challenge in today's synchrotron and free electron laser light sources. This is particularly the case for LCLS II project with FEL beam power up to 200 W in average, and 200 GW in peak. To cover a large photon energy range, the length of an X-ray mirror is often longer than the beam footprint length for much of the applicable energy range. To limit thermal deformation of such a water-cooled X-ray mirror, we propose a technique using side cooling with a cooled length shorter than the beam footprint length. This cooling length can be optimized by using finite element analysis (FEA). Furthermore, we present a second, alternative technique, based on a similar principle; we propose to use a long, single, full-length cooling block on each side of the mirror, and add electric heaters between the cooling blocks and the mirror substrate. The electric heaters consist of a number of cells, located along the mirror length. The total effective length of the electric heater can then be adjustable, by choosing which cells to energize, using electric power supplies. The residual height error of the LCLS-II K-B mirrors, due to Free Electron Laser (FEL) beam heat load, can be reduced to a factor of 10 below the requirement. The proposed techniques are also effective in reducing thermal slope errors, and are therefore applicable to white beam mirrors in synchrotron radiation beam lines.

Towards 10 meV Resolution for Soft X-ray Resonant Inelastic Scattering: The Optical Design of the SIX Beamline and Spectrometer

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Soft x-ray resonant inelastic x-ray scattering (RIXS) has proven to be a valuable tool for studying the electronic structure and low energy excitations in various kinds of materials. The advantages of RIXS encompass several key features such as element and orbital specificity, charge neutrality, and finite cross sections for dipole-forbidden transitions. The advent of the AXES spectrometer at the ESRF (c 1995) and its higher resolution successor SAXES (c 2006) at the Swiss Light Source has proven the merit of these spectrometers and the potential of the technique. This is further evidenced by the construction of larger, high-resolution spectrometers at new RIXS beamlines in various light sources such as the ESRF (ID32), Diamond Light Source (I21), MAX IV (VERITAS), Shanghai Synchrotron Radiation Facility, Taiwan Photon Source. The goal of these beamlines is to push the resolution below the current state of the art (~100 meV at 1000 eV) in order to study very low energy excitations in matter, such as magnetic excitations in condensed matter systems. However, decreasing the bandwidth results in a variety of optical, mechanical, and count rate challenges that are not best addressed merely by a scaling up of current designs. We have chosen for the RIXS spectrometer a Hettrick-Underwood design. We will discuss the design of the RIXS instrument SIX at NSLS-II chosen to deliver a usable count rate at an ultrahigh resolution of 10 meV on both the beamline and spectrometer.

Recent Developments in SRW and Other Open Source Software for X-ray Optics

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Physical-optics simulation of emission, transport and scattering by samples of both fully- and partially-coherent X-ray radiation is required for beamline designers and users at modern Light Sources, including low-emittance storage rings, Energy-Recovery Linacs and Free-Electron Lasers (FEL). The open source "Synchrotron Radiation Workshop" (SRW) software [1,2] is being further developed to meet these needs. We will present recent advances in the following areas: a) reliability and robustness of the fully-coherent wavefront propagation methods; b) efficiency of time- and frequency-dependent wavefront propagation for FEL sources; c) partially-coherent wavefront propagation for storage ring sources; d) treatment of various optical elements and experimental samples; and e) time- and frequency-domain near-field single-electron synchrotron radiation calculations for relevant magnetic fields. We are also implementing virtual X-ray beamlines for the National Synchrotron Light Source II (NSLS-II) [3] and the SLAC Linac Coherent Light Source (LCLS) [4], which will be used to benchmark SRW simulations with experimental data, to facilitate the real beamline commissioning, and will also be made cloud-accessible to interested scientists through a secure IPython interface. Our effort includes the development of a larger open source development environment to support a "spectrum" of scientists from experts in computational X-ray optics to those in other fields who plan to use X-ray beamlines at Light Sources. We plan to support the SHADOW code [5,6] in the near-term and other X-ray codes in the long-term. We will present interactive JavaScript visualizations of SRW data in an IPython-generated browser. We are planning to collaborate with teams pursuing similar developments at SLAC, Advanced Photon Source, European Synchrotron Radiation Facility and European X-FEL.

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X-ray Absorption Spectroscopy under Extremes

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The scanning EXAFS beamline BM23 and the Energy Dispersive X-ray Absorption Spectroscopy (EDXAS) beamline ID24 have been recently completely reconstructed to create a facility dedicated to time resolved and extreme conditions XAS (UPBL11 project).

BM23 is the ESRF standard energy scanning spectrometer dedicated to general purpose XAS. A large part of the beamline activity is dedicated to high pressure. A micro-XAS station has been implemented providing an X-ray focal spot of 3x3 μm^2. The large focal distance allowing the installation of various sample environments and the possibility to record complementary diffraction pattern make this station ideally suited for high pressure XAS studies using the Diamond Anvil Cell (DAC).

On ID24 the original EDXAS scheme allows to record a full XAS spectrum in a single detector acquisition. Coupled to a high flux undulator source, it is a powerful tool to study matter under extreme conditions in particular when short integration times are required – Examples are studies of chemical reactions or of melts stability at very high pressure and temperature. The beamline is equipped with a new high pressure laser heating facility able to reach the multi-megabar regime with temperatures up to 4000 K.

Finally, the combination of high flux with the EDXAS scheme opens on ID24 the possibility to study extreme conditions that can be obtained only in pulsed mode. For example, we have recently obtained good quality EXAFS data on dynamically compressed Fe up to ~ 5.5 Mbars and 12000 K using laser shock compression

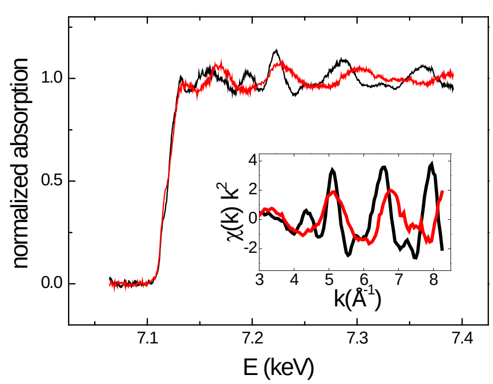

Figure 1: XAS data acquired on ID24 using a unique 100 ps bunch on a laser shock dynamically compressed Fe target

XPD: In-situ, Modulation-enhanced, and Time-resolved X-ray Powder Diffraction at NSLS II

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X-ray Powder Diffraction (XPD) is a damping wiggler beamline at NSLS II designed to collect structural data at high energies, offering exceptional diffraction capabilities such as fast (sub-second) and high angular resolution on the same instrument. The key scientific goals are to structurally characterize materials at the frontier of complexity, whether the frontier is disordered or amorphous structures, nanostructures, heterogeneous materials, or *in situ* environments. In addition to X-ray Powder diffraction (XRD) with high photon flux (time-resolved and in-situ) or with high angle resolution, XPD also offers other techniques such as Pair Distribution Function analysis (PDF), Medium Angle X-ray Scattering (MAXS), Stress-strain scanning, Depth-resolved XRD [1], and Modulation Enhanced XRD (MED) [2]. This suite of structural characterization techniques enables XPD to address future scientific challenges in areas such as hydrogen storage, CO_2 sequestration, advanced structural ceramics, catalysis, and advanced materials synthesis and processing. The science focus of XPD is to characterize such complex systems *in-situ* or *operando*.

This presentation will show the design and optical parameters as calculated [3] and as measured to optimize the use of the above mentioned characterization techniques. XPD major parameters are energy tunability (between 30 and 70 keV), high flux ($\sim 2 \times 10^{13}$ p/s at 50 keV and 500 mAmp), high resolution ($\Delta E/E \sim 10^{-3}$ - 2×10^{-4}), and adjustable beam size (50 µm (vert) x 500 µm (horiz) to mm size) [4]. The above parameters are achieved by using a sagittally bent double Laue monochromator, whose concept was developed in house [5, 6], a large (1.42 m) dynamically bent Pt coated Si mirror and a high resolution channel-cut monochromator. We will also provide a few examples of the scientific measurements that were recently performed during the science commissioning of the beamline.

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Synchronizing MEMS-Based X-Ray Optics to Storage-Ring Fill Patterns

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Dynamic, MEMS-based optics are able to temporally modulate synchrotron x-ray beams[1]. Such x-ray optics consist of an oscillating x-ray micromirror that can diffract x rays with nearly 100% efficiency; a micrograph of a device is shown in Fig. 1. The device is positioned to the diffraction condition and then set to oscillate at rates up to ~100 kHz; x rays are transmitted only when an x-ray pulse is incident and the crystal is rotating thru the diffraction condition. The timing gap between transmitted pulses, T_g, can be varied by adjusting the angle of the device relative to the incident beam ($\Delta\theta$ in Fig. 2). However, the probability that an x-ray bunch will arrive while the device is within its diffraction condition is fairly low, for a device operating at an arbitrary frequency. Here we present an optic which can be synchronized to the frequency of the APS accelerator to reproducibly select x rays from a given bunch. We quantify the improvements in x-ray throughput that a synchronized device provides and show the x-ray diffractive time window to be on the order of a few nanoseconds. Synchronization of x-ray micromirrors is an important step toward the application of these devices to the synchrotron environment.

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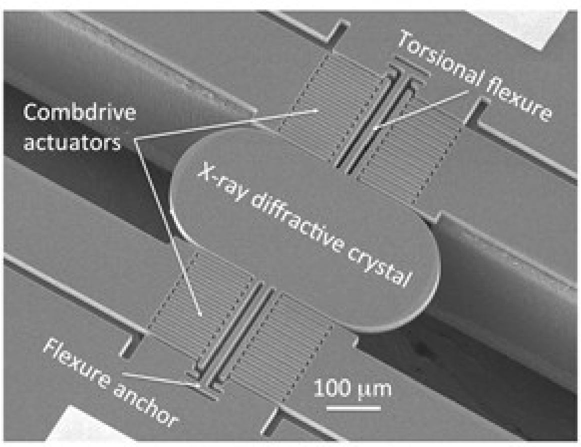

Fig. 1. Micrograph of a MEMS diffractive optic. The x-ray beam is incident from the lower right and diffracts to the upper left.

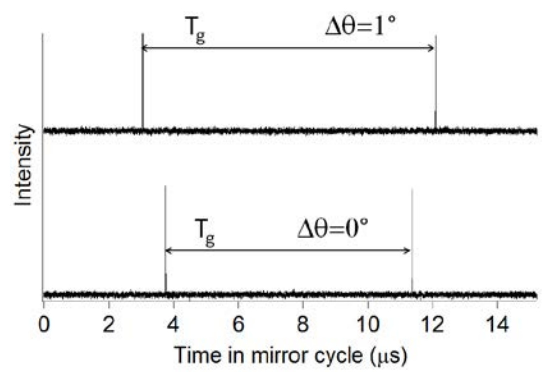

Fig. 2. The oscillating MEMS optic only transmits x rays when the rotating crystal satisfies the diffraction condition; the diffraction-time windows are on the order of a few nanoseconds. The data shown here demonstrate that the timing of this condition can be varied by adjusting the angle of the device relative to the incident beam.

Single-shot Femtosecond X-ray Streaking Method for Ultrafast Dynamics

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The advent of ultrashort and intense x-ray pulses from free electron lasers has paved the way to new possibilities for sub-picosecond time-resolved studies. Typically these studies are carried out in a pump-probe method where a single probe beam is directed to the sample at a fixed delay time after the pump beam. This requires multiple pump-probe cycles at different delays in order to reconstruct the full dynamics. To circumvent this problem we have developed and demonstrated unique grating-based setups that allow for recording the full dynamics of transient phenomena, with femtosecond time resolution, and in single-shot manner[1].

We present two independent implementation of this scheme in the XUV energy range (performed at FLASH, DESY) and in the multi-keV range (recently performed at LCLS, SLAC). The accessible time window is 1.57ps for 60eV at FLASH, and 350fs for 5050eV at LCLS. We will also highlight the key features of the setup, including the robustness of the diffraction gratings with respect to beam damage and misalignment, and the absence of timing jitter between the probe beams due to their fixed geometrical arrangement.

At last, we show measurements of ultrafast demagnetization dynamics of ferromagnetic layers (in the XUV range), and of time resolved Bragg reflectivity of inorganic and macromolecular crystals (in the multi-keV range).

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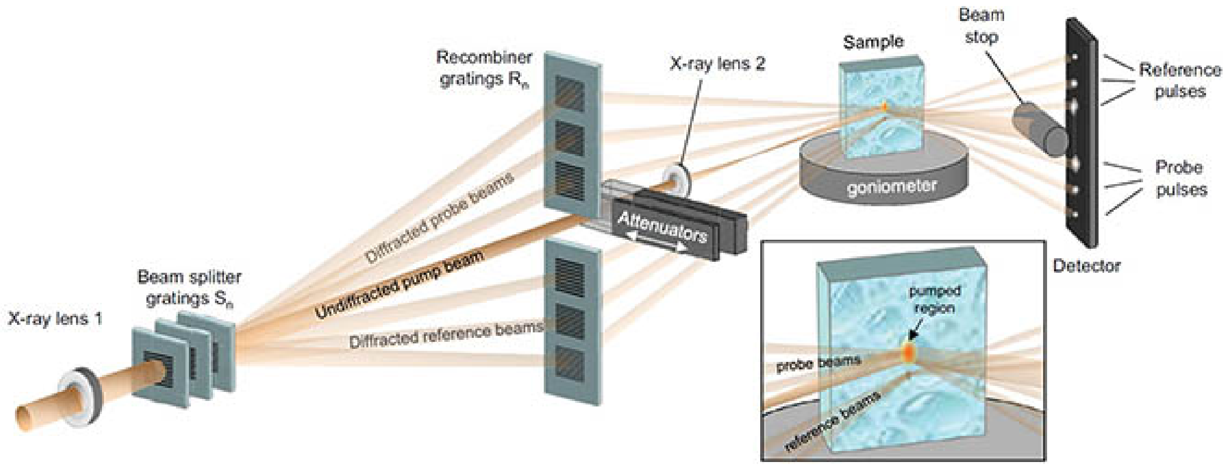

Schematic drawing of the LCLS experiment setup. The multiple probe beams created by the upstream splitter gratings are re-directed with the recombiner gratings such that they follow the direct (transmitted) beam after controlled delay times, all from a single pulse. Only 3 different delay channels are shown for simplicity, the experimental implementation featured more than 10 channels.

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Commissioning of the Femto-Slicing Project at Synchrotron SOLEIL

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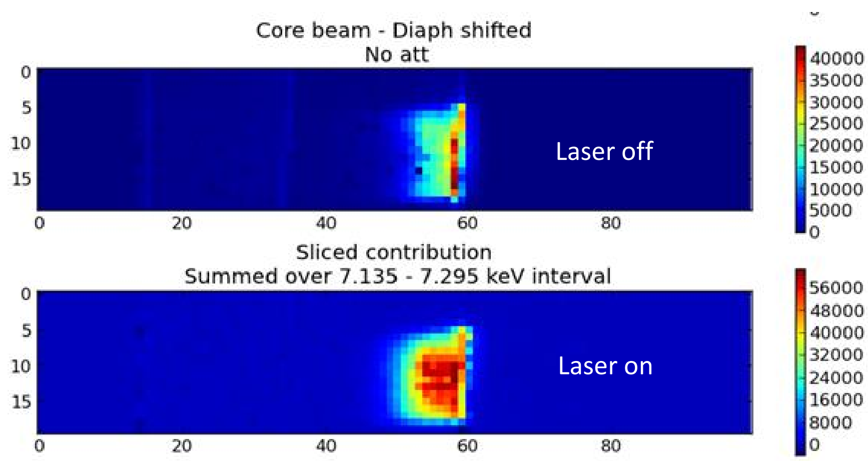
The Femto-slicing project at SOLEIL is currently under commissioning. It will enable to serve several beamlines with sub-ps pulses of soft and hard X-rays for pump-probe experiments, with reasonable flux and with a 1-10 kHz repetition rate. It is based on the interaction of a femtosecond Ti:Sa laser with electrons circulating in the magnetic field of a modulator wiggler, that provides the electron beam energy modulation on the length scale of the laser pulse [1]. The laser system is configured at a 1 kHz repetition rate for delivering pulses of about 5 mJ with pulse duration at the compressor output of about 25 fs. The same laser is used to both create the slicing process and to pump the experiments [2]. This layout yields natural synchronization between IR laser pump and X-ray probe pulses, only affected by jitter-drift associated with beam transport. The first laser-electron interaction was observed at the end of 2014 by detecting the coherent THz radiation induced. In February 2015, the first sub-ps hard X-rays pulses were detected on the CRISTAL beamline with a 2D XPAD detector located behind the monochromator. Presently, we are optimizing the interaction process and photon flux on the hard X-rays CRISTAL beamline versus laser and electron beams parameters. In parallel, we are installing the equipment in the machine and on the laser transport to serve by the end of 2015, the soft X-rays TEMPO beamline. In this paper, we present the results of the optimization, from the laser alignment in the interaction section observed through an Infra-Red and Terahertz (THz) diagnostics, to the comprehensive study of the various femtoslicing parameters in terms of THz emission, and photons flux on the CRISTAL beamline. The results are compared with predicted values based on electron tracking and photon optics calculations.



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Soft X-ray Excitation-emission Matrix Measurement and Analysis

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Soft x-ray excitation-emission matrices (EEMs) are measurements of x-ray fluorescence and scattering intensities over a wide range of incident and emission energies. They provide a holistic approach for the characterization of materials that is sensitive to composition, chemistry and physical structure. At the Spherical Grating Monochromator (SGM) beamline at the Canadian Light Source, EEMs are recorded by measuring emission spectra with an array of specifically positioned (with respect to the beam polarization and scattering vector) soft x-ray compatible silicon drift detectors while scanning the incident x-ray energy over a wide energy range. Soft x-ray energies between 250 and 2000 eV are used to access light element K edges as well as the L, M and N edges of heavier elements is possible. In addition, high resolution, first order diffraction peaks from structures with a d-spacing between 0.3 nm to 4 nm are observed. The analysis of EEMs from reference minerals, soils and sediments has been performed and demonstrates the ability for this technique to probe the chemical and structural relationships between all major soil components.

Momentum-Resolved Resonant Inelastic X-Ray Scattering endstation (qRIXS) at the Advanced Light Source

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Momentum-resolved resonant inelastic soft X-ray scattering spectroscopy (qRIXS) reveals the energy and momentum dependence of a wide range of many-body collective modes [1], and has been identified as one of the most promising techniques for studying correlated electron systems. An endstation (qRIXS) is currently under construction, and will soon be available for such studies at the ALS. Multiple modular high resolution/high throughput soft X-ray emission spectrographs coupled to a rotating chamber will allow the X-rays scattered from the sample to be measured at different emission angles relative to the incident photon beam, thus realizing a true momentum-resolved capability. Furthermore, its compact design enables roll-up operation at different facilities, notably at the Linac Coherent Light Source (LCLS) of SLAC National Accelerator Laboratory for time & momentum-resolved RIXS measurements. I will discuss the instrument and a few key experiments that can be performed with it at the ALS and/or LCLS.

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New Perspectives in Inelastic X-ray Scattering – UPBL6@ID20

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In the frame of the ESRF Upgrade Phase I, the UPBL06 project aimed at the construction of a state-of-the-art hard inelastic x-ray scattering (IXS) beam line on undulator port ID20. The beam line is operational since October 2013, and is fully dedicated to the study of electronic and magnetic excitations in condensed matter. Scientific goals comprise the investigation of strongly correlated electron systems, functional materials, and chemical reactions in liquids and gases. The beam line hosts two spectrometers: one dedicated to resonant IXS studies with variable energy resolution down to 25 meV (at 11 keV), and one optimized for investigations by x-ray Raman scattering, equipped with six modules of 12 spherically bent crystal analysers each, covering a very large solid angle in both the horizontal and vertical scattering planes. The use of hard X-rays ensures bulk sensitivity and compatibility with complex sample environments, such as chemical reactors and diamond anvil cells for *operando* experiments or under extreme conditions. We will briefly outline the optical concept of the beam line, present the key characteristics of the two spectrometers, and illustrate the current capabilities by the most challenging experiments, recently performed on the instruments.

High Resolution Resonant Inelastic X-ray Scattering: First Results and Opportunities

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Resonant inelastic x-ray scattering (RIXS) has proved to be a versatile tool to investigate elementary excitations in correlated electron systems. In RIXS processes, a core hole is created by absorbing a photon with well-defined energy and momentum. In the presence of this core potential, the system undergoes a series of excitations [1]. So far, charge-transfer and dd excitations have been successfully measured[2]. In recent years, with improving energy resolutions, magnetic excitations are also been detected[3], [4]. With the unparalleled energy resolution we achieved at the Cu K-edge (25 meV)[5], it is possible to detect the lattice excitations with RIXS.

RIXS phonon spectra fundamentally differ from the inelastic neutron and x-ray scattering methods. The lattice responds to the sudden change in charge density caused by the absorbed photon. This response manifests itself with phonon modes, the intensity of which reveals the electron-phonon coupling strength[6], [7]. We will present the novel instrument developed for high resolution RIXS measurements, and report our first results.

This instrument is part of the new inelastic x-ray scattering beamline at PETRA-III synchrotron in Hamburg, Germany. In addition to RIXS, the beamline features a non-resonant inelastic x-ray scattering instrument for x-ray Raman scattering measurements. We will also briefly present the capabilities of this new facility, which is starting general user operation in May 2015.

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The Ultrahigh Resolution Inelastic X-ray Scattering (IXS) Beamline at NSLS-II: First Results*

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This contribution will present the final design and implementation of the ultrahigh resolution inelastic x-ray scattering (IXS) beamline at NSLS-II (Figure 1), which has a design goal of sub-meV to the ultimate 0.1 meV resolution for inelastic x-ray scattering experiments on fast dynamics in exotic material systems ranging from soft matter, colloids, and biological materials with complexity and disorders in mesoscopic length scales, to systems in confined geometries such as surfaces, interfaces and in extreme pressure and temperature. The key instrument is a novel spectrometer with analyzer optics based on a highly-dispersive back-reflection optical system on a 5m scattering arm that covers a wide range of momentum transfer. Performance results of the instrument based on technical commissioning and early science experiments will be presented.

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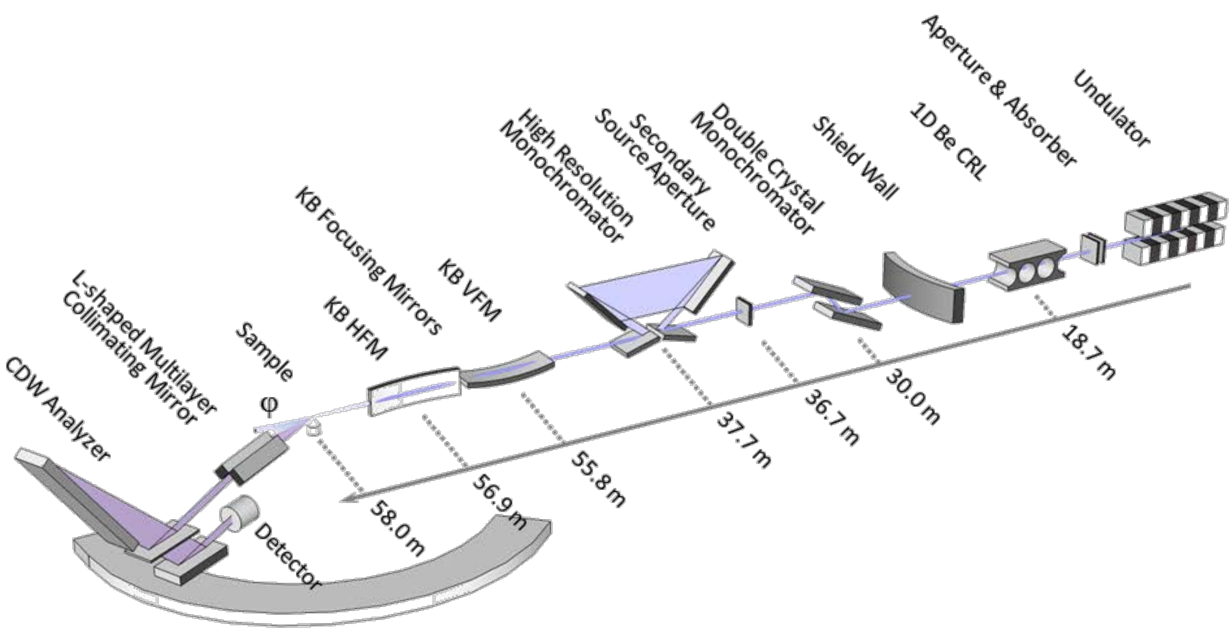

Figure 1. Optical layout of the ultrahigh resolution inelastic x-ray scattering (IXS) beamline as currently implemented at NSLS-II to realize sub-meV resolution initially, with provision for improving to the ultimate goal of 0.1 meV.